

WADC-TR-53-373

Suppl 9

A00292152

PROPERTY OF THE U. S. AIR FORCE
Air Force Institute of Technology
Library

A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

Donna J. Tate

TECHNICAL REPORT NO. WADC-TR-53-373 Suppl 9

November 1962

Directorate of Materials and Processes
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

Statement A
Approved for Public Release

20040805003

BEST AVAILABLE COPY

NOTICES

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Qualified requesters may obtain copies of this report from the Armed Services Technical Information Agency, (ASTIA), Arlington Hall Station, Arlington 12, Virginia.

This report has been released to the Office of Technical Services, U.S. Department of Commerce, Washington 25, D.C., in stock quantities for sale to the general public.

Copies of this report should not be returned to the Aeronautical Systems Division unless return is required by security considerations, contractual obligations, or notice on a specific document.

FOREWORD

This report was prepared by Mrs. Donna J. Tate, Office of the Director, Directorate of Materials & Processes, Deputy for Technology, Aeronautical Systems Division, Wright-Patterson AFB, Ohio.

Technical reports published by the Directorate of Materials and Processes during the period 1 July 1961 - 30 June 1962 are abstracted herein. Reports on research conducted by Directorate of Materials & Processes personnel as well as that conducted on contract are included.

Qualified requestors may obtain copies of the reports abstracted herein direct from the Armed Services Technical Information Agency, (ASTIA), Arlington Hall Station, Arlington 12, Virginia. ASTIA's services for Department of Defense contractors are available through the Field of Interest Register on a Need to Know, certified by the cognizant military agency or their project or contract.

Abstracts which indicate "OTS Release" may be obtained, by the general public, from the Office of Technical Services, 1200 S. Eads St., Arlington, Va.

ABSTRACT

These reports cover basic and applied research in the materials area being conducted by the Metals and Ceramics, Non-Metallic Materials, Physics, Manufacturing Technology, and Applications Laboratories of the Directorate of Materials and Processes.

This report has been reviewed and is approved.



A. M. LOVELACE
Technical Director
Directorate of Materials &
Processes
Deputy for Technology

TABLE OF CONTENTS

I. Technical Reports and Technical Notes 1 July 1960 - 30 June 1961

	Page
Ceramics, Cermets, and Graphite -----	1
Ceramics and Cermets -----	1
Graphite -----	6
High Strength Metals -----	11
High Temperature Metals -----	14
Physical Metallurgy -----	18
Mechanical Metallurgy -----	29
Joining -----	54
Non-Destructive Testing -----	57
Coatings -----	59
Ceramic -----	59
Organic -----	60
Refractory Metal -----	63
Corrosion -----	66
Polymers and Synthesis Studies -----	70
Elastomers -----	92
Composites -----	100
Erosion -----	107
Plastics -----	108
Fibrous Materials -----	119
Fuels, Lubricants and Fluids -----	130

TABLE OF CONTENTS (Continued)

	Page
Hydraulic Fluids -----	136
Heat Transfer Fluids -----	138
Lubricants -----	140
Solid Film Lubricants -----	153
Electrical & Electronic Materials -----	157
Nuclear Measurements -----	167
Thermo-Physics -----	169
Analytical Techniques -----	183
Design Criteria -----	192
Application Studies -----	204
Electronics -----	208
Chemical Engineering -----	210
Fabrication -----	213
Basic Industry -----	227
II. Investigator Index -----	231
III. Contractor Index -----	247

I. Technical Reports and Technical Notes 1 July 1961 -
30 June 1962

CERAMICS, CERMETS, AND GRAPHITE

CERAMICS AND CERMETS

WADC TR 59-316, Part III, OTS Release

February 1962

SUBJECT: MECHANISM OF WEAR OF NON-METALLIC MATERIALS
INVESTIGATOR: C. H. Riesz, H. S. Weber
CONTRACT: AF33(616)-6920, Armour Research Foundation
ABSTRACT: Friction and wear of single-crystal sapphire sliding under 10^{-3} mm Hg vacuum from 30 to 1550°C were studied. Surface cleanliness was of prime importance. High coefficients of friction and stick-slip sliding occurred at and below 300°C. From 300 to 1000°C lower coefficients and smooth sliding occurred. Above 1000°C, high stick-slip appeared, but at 1350 and 1550°C less stick-slip and lower coefficients dominated.

Chevron-shaped subsurface fractures of the sapphire plate was observed in the wake of the sapphire slider at and below 300°C. Their formation was orientation dependent and occurred when the C_o -axis of the plate was inclined 86-89° to the surface, opposite to the direction of sliding. In the absence of fracture, extensive adhesions were noted.

Under high vacuum, a weld-adhesion mechanism of sliding friction seems useful, especially above 1000°C. Below 300°C, it may be valid only if clean surfaces are in sliding contact; molecular forces influence sliding friction and may cause strong adhesions.

WADC TR 59-602, Part II, OTS Release

February 1962

SUBJECT: INVESTIGATION OF MATERIALS CAPABILITIES
OF MATERIAL SYSTEMS IN SOLID ROCKET
MOTORS. PART II. Analysis of Heat
Transfer Factors
INVESTIGATOR: E. M. Sadownik
CONTRACT: AF33(616)-7365, Aerojet-General Corp.
ABSTRACT: Temperature histories of various nozzle
materials systems were analyzed parametrically, and a series

Manuscript released October 1962 as a WADC Technical Report

WADC TR 59-602, Part II (Continued)

of hot-flow tests were conducted in support of the analytical study. Results of the analyses showed the interrelationship of material combinations in relation to duration. Results of the test program showed (1) effects of flame barrier and insulation thickness on duration capability, (2) the proximity of actual to calculated temperature distributions, and (3) the effect of aluminum oxide deposition on materials system capability.

WADC TR 60-184, Part II

September 1961

SUBJECT: EFFECT OF BASIC PHYSICAL PARAMETERS ON
ENGINEERING PROPERTIES OF INTERMETALLIC
COMPOUNDS

INVESTIGATOR: J. H. Westbrook, D. L. Wood

CONTRACT: AF33(616)-6144, General Electric Research
Laboratory

ABSTRACT: The tensile behavior of the CsCl structure
compound AgMg are extensively documented in terms of strain,
strain rate, temperature, grain size, composition, and metal-
lurgical processing treatment. Three regimes of deformation
behavior are observed: low temperatures, intermediate tempera-
tures, and high temperatures. In each of these regimes dif-
ferent deformation processes operate. Specific findings
have practical as well as scientific import. Certain ex-
ploratory studies on other intermetallics are also reported.

WADD TR 60-782, Part V, OTS Release

November 1961

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT
HIGH TEMPERATURE PART V. Mass Spectro-
metric Study of Gaseous Molecules Above
the AgSn, AuSn, and CuSn Alloys

INVESTIGATOR: M. Ackerman, J. Drowart, F. Stafford,
G. Verhaegen

CONTRACT: AF61(052)-225, University of Brussels

ABSTRACT: The molecules AgSn, AuSn, and CuSn have
been identified. Dissociation energies have been measured
to be:

$$D_0^{\circ}(\text{Sn}_2) = 45.8 \pm 4 \text{ kcal/mole}$$

$$D_0^{\circ}(\text{AgSn}) = 31.6 \pm 5 \text{ kcal/mole}$$

$$D_0^{\circ}(\text{AuSn}) = 57.5 \pm 4 \text{ kcal/mole}$$

$$D_0^{\circ}(\text{CuSn}) = 41.4 \pm 4 \text{ kcal/mole}$$

November 1961

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE PART VI. Studies of the Vapors of the Systems Au-Cr and Au-Pd by Mass Spectrometry

INVESTIGATOR: M. Ackerman, F. E. Stafford, G. Verhaegen

CONTRACT: AF61(052)-225, University of Brussels

ABSTRACT: The molecules Au-Cr and Au-Pd have been identified. From the data for the reaction $Au_2 + X = AuX + Au$, and $D_0^{\circ}(Au_2) = 51.5$ kcal/mole:

$$D_0^{\circ}(AuCr) = 50.4 \pm 3.5 \text{ kcal/mole}$$

$$D_0^{\circ}(AuPd) = 33.3 \pm 5 \text{ kcal/mole}$$

April 1961

SUBJECT: INVESTIGATION OF INTERMETALLIC COMPOUNDS FOR VERY HIGH TEMPERATURE APPLICATIONS

INVESTIGATOR: J. Booker, R. M. Paine, A. J. Stonehouse

CONTRACT: AF33(616)-6540, Brush Beryllium Co.

ABSTRACT: Intermetallic beryllides from the systems tantalum-beryllium, tungsten-beryllium, and hafnium-beryllium along with the di-silicides of tungsten, tantalum, and molybdenum, were screened for compounds capable of serving as structural materials at temperatures above 2500°F. The compounds studied were $TaBe_{12}$, Ta_2Be_{17} , Hf_2Be_{21} , $MoSi_2$, $TaSi_2$, and WSi_2 . The preparation, fabrication, oxidation resistance, and thermal-shock resistance are discussed. Values are given for the transverse-rupture strengths, impact resistance, mean-linear coefficients of thermal expansion, enthalpy, specific heat, and thermal conductivity.

An investigation of the rates of oxidation of intermetallic beryllides was initiated. The oxidation of $TaBe_{12}$, Hf_2Be_{21} , $ZrBe_{13}$, and Ta_2Be_{17} in the range 2300° to 2750°F was found to obey an exponential rate law which was cubic or a higher power rate law. In most cases, the cubic rate law applied. The products of the oxidation of $ZrBe_{13}$ at 2500°F were identified as Zr_2Be_{17} and BeO . Tentative activation energies for a cubic rate process were calculated for $TaBe_{12}$ and Hf_2Be_{21} .

June 1961

SUBJECT: ORDERING IN OXIDE SOLID SOLUTIONS
INVESTIGATOR: H. H. Wilson
CONTRACT: AF33(616)-6870, Ceramic Engineering Dept.,
Clemson College
ABSTRACT: A study was made of solid solutions formed from mixtures of magnesium oxide and either with manganese, iron, cobalt, or nickel oxide to determine if, by suitable heat treatment, ordering could be induced in certain selected compositions. Heat treatment consisted of firing the prepared solid solutions in suitable atmospheres at 200° intervals from 200°C to 1200°C. The specimens were held at each temperature for 100 hours and analyzed by x-ray diffraction to detect superlattice formation.

No superlattices were found in the samples. It is possible that the lack of ordering might be due to either insufficient time at the proper temperature or to the maximum temperature being too low to allow the required ionic diffusion. Future work will include the use of extended times and higher temperatures. Also, the use of mechanical stress to enhance diffusion will be investigated.

July 1961

SUBJECT: SURFACE AND ENVIRONMENTAL EFFECTS ON
CERAMIC MATERIALS
INVESTIGATOR: P. Gibbs, G. S. Baker, B. G. Dick, Jr.,
E. K. Beauchamp, M. H. Miles, W. G.
Rogers
CONTRACT: AF33(616)-6832, University of Utah
ABSTRACT: Polycrystalline sintered compacts of Alucor MC alumina doped with $\text{MgO}(\text{CO}_3)_2$ have been deformed in three point beam loading in the temperature range of 1000° to 1350°C. It is suggested that polycrystalline creep is controlled by diffusion of vacancies in the steady state. Creep behavior of Linde corundum 0.1 inch diameter rods in three point loading (1000°-1250°C) is described. At the lowest temperatures and stresses studied, measurable creep began after about an hour's time delay, and increased at an accelerating rate. A study is described of the dispersal of impurities from a metal contact over the free surface of corundum at 10^{-4} mm. Hg, by measuring the current flowing between two vacuum deposited Ag films as a function of time, temperature (20°-640°C), and applied field (0-12v.). It is suggested that detaching Ag from the deposited film controls

ASD TR 61-182 (Continued)

the rate of formation of a "semi-conducting bridge" on or just beneath the corundum "free surface".

WADD TR 61-262

June 1961

SUBJECT: INVESTIGATION OF SINTERABLE OXIDE POWDERS AND CERAMICS MADE FROM THEM

INVESTIGATOR: C. Hyde, W. Duckworth

CONTRACT: AF33(616)-262, Battelle Memorial Institute

ABSTRACT: This program is concerned with the strength of nonporous monophase ceramics as a function of their microstructure, and with the basic nature of sinterable powders. Sinterable powders provide a convenient way to study a wide variety of microstructural effects without introducing variations in density or purity. MgO powders, prepared by calcining a high-purity basic magnesium carbonate, were studied as representative examples of sinterable powders. The temperature of calcination of the carbonate was found to have a critical effect on the density that could be obtained in sintered compacts of the oxide. The density of compacts of each calcine increased during sintering until a ceiling was reached after which grain growth occurred without further densification. Ceiling densities of 97 to 98% of theoretical were attained. Sintering in various atmospheres or for moisture-free atmosphere reduced the temperature at which the ceiling density was reached. Indications were that sintering atmosphere affected grain growth. The work demonstrated the importance of controlling processing variables to assure uniformly reproducible specimens for strength measurements.

ASD TR 61-350

November 1961

SUBJECT: INVESTIGATION OF SINGLE-CRYSTAL AND POLYCRYSTALLINE TITANIUM DIBORIDE: METALLOGRAPHIC PROCEDURES AND FINDINGS

INVESTIGATOR: C. T. Lynch, F. W. Vahldiek, S. A. Mersol, C. R. Underwood

ABSTRACT: Single-crystal and polycrystalline TiB_2 was examined to develop applicable metallographic techniques for sectioning, mounting, grinding, polishing, and etching of TiB_2 specimens. This work demonstrates the usability and practicability of using various SiC papers together with different grades of diamond paste or polishing wheels in preference to using cloths. It has been found that H_2SO_4 , as a constituent of etchants, produces more reliable and

WADC TR 53-373 Sup 9

5

ASD TR 61-350 (Continued)

more consistent results than HF. The same is true of H_2SO_4 when used as a constituent of electrolytes. Single-crystal TiB was found to have a type of Widmanstätten Structure while polycrystalline TiB₂ had a "needle-like" pattern.

GRAPHITE

WADD TR 60-782, Part III

November 1961

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE PART III. Mass Spectrometric Studies of the Molecules BC₂ in the Vapor Above the System Boron-Carbon

INVESTIGATOR: G. Verhaegen, F. E. Stafford, M. Ackerman

CONTRACT: AF61(052)-225, University of Brussels

ABSTRACT: The molecules BC₂ and B₂C have been identified in the vapor effusing from graphite Knudsen cells containing boron. The atomization energy $D_0(B-C-C) = 297 \pm \text{kcal/mole}$.

WADD TR 60-782, Part VI

November 1961

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE PART VI. Studies of the Vapors of the Systems Au-Cr and Au-Pd by Mass Spectrometry

INVESTIGATOR: M. Ackerman, F. E. Stafford, G. Verhaegen

CONTRACT: AF61(052)-225, University of Brussels

ABSTRACT: The molecules Au-Cr and Au-Pd have been identified. From the data for the reaction $Au_2 + X = AuX + Au$, and $D_0(Au_2) = 51.5 \text{ kcal/mole}$:

$$D_0(AuCr) = 50.4 \pm 3.5 \text{ kcal/mole}$$

$$D_0(AuPd) = 33.3 \pm 5 \text{ kcal/mole}$$

WADD TN 61-18

April 1961

SUBJECT: RESEARCH AND DEVELOPMENT ON ADVANCED GRAPHITE MATERIALS

INVESTIGATOR: R. Wilson, C. A. Douglass

CONTRACT: AF33(616)-6915, Union Carbide Corp.

WADC TR 53-373 Sup 9

WADD TN 61-18 (Continued)

ABSTRACT: A review is given of results from activities from 1 May 1960 through 15 October 1960, on a three year United States Air Force - Department of Defense Research and Development contract to develop improved materials in the graphite and carbon field specifically for missile and astronautics applications. Progress is reported both on new grades of graphite which extend the range of physical properties and on narrowing the spread of properties within a given grade. Preliminary data are included on numerous physical properties of the various graphite materials.

WADD TR 61-72, Vol I

September 1961

SUBJECT: RESEARCH AND DEVELOPMENT ON ADVANCED GRAPHITE MATERIALS VOL I. Observations by Electron Microscopy of Dislocations in Graphite

INVESTIGATOR: R. Sprague

CONTRACT: AF33(616)-6915, National Carbon Company

ABSTRACT: This report describes the application microscope to the study of dislocations in graphite. Sample preparation is detailed. Photographs are shown illustrating the wide variety of dislocation patterns observed and some of the information obtained from them is discussed.

WADD TR 61-72, Vol III, OTS Release

February 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON ADVANCED GRAPHITE MATERIALS VOL III. Decoration of Dislocations and Low Angle Grain Boundaries in Graphite Single Crystals

INVESTIGATOR: R. Bacon, R. Sprague

CONTRACT: AF33(616)-6915, National Carbon Co.

ABSTRACT: Dislocations and low angle grain boundaries in single crystal graphite have been decorated by introducing into the crystal Cs, Br, and FeCl_3 , which are known to form lamellar compounds with graphite. The existence of pockets or internal cracks, initiated at and bounded by tilt boundaries has been inferred. Circular pockets of trapped gas bubbles are believed to nucleate at sites of condensed vacancy disks.

WADD TR 61-72, Vol IV, OTS Release

October 1961

SUBJECT: RESEARCH AND DEVELOPMENT ON ADVANCED

WADD TR 61-72, Vol IV (Continued)

GRAPHITE MATERIALS VOL IV. Adaptation
of Radiographic Principles to the Quality
Control of Graphite

INVESTIGATOR: R. W. Wallouch
CONTRACT: AF33(616)-6915, Advanced Materials Laboratory
ABSTRACT: Special equipment and techniques were developed for the radiographic inspection of large size graphite shapes, as well as for complex multi-thickness shapes. Because of the low atomic number of the element carbon, techniques are used which discriminate between the transmitted and scattered radiation. Various methods are described which have been used for nondestructively testing graphite and exposure charts relating exposure time to graphite thickness for various source voltages were prepared.

WADD TR 61-72, Vol V, OTS Release

July 1961

SUBJECT: RESEARCH AND DEVELOPMENT OF ADVANCED
GRAPHITE MATERIALS VOL V. Analysis of
Creep and Recovery Curves for ATJ
Graphite

INVESTIGATOR: E. J. Seldin, R. N. Draper
CONTRACT: AF33(616)-6915, National Carbon Co.
ABSTRACT: Flexural creep tests have been made on specimens of ATJ graphite at temperatures ranging from 2300°C to 3000°C. The creep curves have been fitted by graphical and analytical means to four equations. Attempts were made to determine the stress and temperature dependence of several of the parameters in the equations.

Of the four equations which were used, one could be related to simple rheological models having some physical significance. A model was found useful not only in describing the creep curves, but also in describing the recovery after creep and relating the behavior in recovery to the behavior during creep. Generally good agreement was found between the qualitative predictions of the models and the observed behavior. Using a model, an activation energy for the steady state creep rate was determined to lie between 70 and 76 kcal/mole. Some deviations between the actual behavior of the material and that predicted by the simple models suggest that some modifications may be made to obtain a better model.

wadd tr 60-143, Part II

May 1961

SUBJECT: RESEARCH STUDY TO DETERMINE THE PHASE
EQUILIBRIUM RELATIONS OF SELECTED METAL
CARBIDES AT HIGH TEMPERATURES

INVESTIGATOR: R. T. Dolloff, R. V. Sara

CONTRACT: AF33(616)-6286, National Carbon Company

ABSTRACT: The work herein is the result of an investigation of phase equilibria in the binary system, tungsten-carbon. A tentative phase diagram is presented which differs significantly from the one proposed by Sykes in 1930 and which is generally accepted today. The data were obtained by high-temperature differential thermal analysis and classical quenching procedures, both supplemented by metallographic, x-ray, and chemical techniques. Results for the tungsten-carbon binary system indicate a eutectic between W and W_2C at $2735^{\circ}C$, and a eutectic between W_5C_3 and C at $2765^{\circ}C$. The W_2C lattice accommodates 72 and 74 atomic per cent W at 2475° and 2735° respectively. Carbon solubility is evident only at $2540^{\circ}C$, the eutectoid temperature. A new phase, W_5C_3 , has been discovered which is stable only above $2540^{\circ}C$. WC decomposes at $2730^{\circ}C$ into W_5C_3 and C.

ASD TR 61-445

October 1961

SUBJECT: THERMODYNAMICS OF INTERSTITIAL SOLID
SOLUTIONS

INVESTIGATOR: L. Kaufman

CONTRACT: AF33(616)-6788, Manufacturing Labs, Inc.

ABSTRACT: The thermodynamics of interstitial solid solutions in terms of the contributions of positional and vibrational entropy and zero point enthalpy has been considered in detail for ideal and restricted interstitial solutions. The results have been applied to calculation of phase equilibria in interstitial iron-carbon alloys at one atmosphere and high pressure. Comparison with observations on kinetics of the bainite reaction and high pressure equilibria action yields good agreement. A method evolved for computing the entropy of solutions and intermetallic compounds from $0^{\circ}K$ to the melting point has been applied to 35 NaCl type compounds (including high melting carbides, oxides and nitrides). These computations compare favorably with the experimental data. A study of the interstitial solutions in the titanium-oxygen system indicates that the high temperature stability of the h.c.p. solution is due to the enthalpy of formation.

ASD TDR 62-88, OTS Release

January 1962

SUBJECT: INVESTIGATION OF MATERIALS FOR VACUUM
INSULATION UP TO 4000°F
INVESTIGATOR: P. E. Glaser, A. E. Wechsler, I. Simon,
J. Berkowitz
CONTRACT: AF33(616)-6816, Arthur D. Little, Inc.
ABSTRACT: This problem encompasses an investigation
of materials for vacuum insulation up to 4000°F. ADL-17,
graphite fibers, thoria powder, and tantalum radiation shields
were chosen as insulation components for long-term high-
temperature tests on the basis of theoretical studies of both
the mechanisms of heat transfer through evacuated insulation
materials and preliminary experimental work. Thermal pro-
tection systems consisting of these materials were chemically
and physically stable enough to be effective insulators for
over 100 hours at temperatures of 3000°F and for several hours
at 3500°F. Although we utilized only readily available
materials, their insulating performance points to the potential
effectiveness of improved materials.

ASD TDR 62-90, OTS Release

February 1962

SUBJECT: SYNTHESIS OF PYROLYSIS OF REFRACTORY
METAL ALKOXIDES
INVESTIGATOR: K. S. Mezdiyasni, C. R. Conners, C. A.
Pratt
ABSTRACT: Metal alkoxydes were prepared based on
the D. C. Bradley, et al procedure. Sufficient quantities
of principally zirconium isopropoxide and tetra-tert butoxide
were produced to use as starting material for impregnation
and coating purposes. The compounds were prepared by simple
chemical reactions but under extremely dry atmosphere.

The purity, concentration, order of
stability, melting temperature, vapor pressure, and decom-
position temperature of the compound are described.

While the compound may be applicable to
coating various materials, to date experiments have been
limited to coating graphite substrates. The compound was
pyrolyzed at 250° - 500°C. Tetragonal-monoclinic thin film
ZrO₂ was deposited on graphites of varying purity.

HIGH STRENGTH METALS

WADD TR 60-893, OTS Release

August 1961

SUBJECT: RESEARCH AND DEVELOPMENT ON HIGH-PRESSURE
HIGH-TEMPERATURE METALLURGY

INVESTIGATOR: L. Kaufman

CONTRACT: AF33(616)-6837, Manufacturing Labs, Inc.

ABSTRACT: The accomplishments of the contract
period 8 October 1959 to 7 February 1961 are summarized.
The experimental investigations on the effect of high hydro-
static pressure on phase transformations in various sub-
stitutional iron-base alloys, including iron-chromium, iron-
nickel, and iron silicon, yield data which are in close
agreement with theoretical prediction. A study of the effects
of pressure on iron-carbon alloys shows a general shift of
the equilibrium phase boundaries to lower carbon contents
and temperatures with pressure. Pressure also acts to re-
tard both the rate of tempering and the isothermal trans-
formation of metastable austenite. The synthesis of f.c.c.
MoC has been carried out at pressures in excess of 35 kilo-
bars at a temperature of 2000°. The thermally activated
recovery and recrystallization processes are shown to be
retarded in a study of copper and 70-30 brass. Experimental
data have been obtained for the effect of pressure on the
Hall voltage of cerium. Preliminary studies have been
carried out on the high pressure sintering of Al₂O₃.

ASD TR 61-386

31 July 1961

SUBJECT: DEVELOPMENT OF NEW AND USEFUL ELEVATED-
TEMPERATURE STEELS FOR AIRCRAFT APPLI-
CATIONS

INVESTIGATOR: A. Kasak, V. K. Chandhok, E. J. Dulis

CONTRACT: AF33(616)-386, Crucible Steel Company
of America

ABSTRACT: This research project was aimed at de-
veloping a new heat-treatable stainless steel with an out-
standing combination of strengths at ambient and elevated
temperatures. Of the large number of experimental com-
positions investigated, the 0.15 C - 14.5 Cr - Mo - V -
Co type steels indicated the best combination of desired
characteristics and properties. Analysis of the effects
of the alloying elements showed that molybdenum is an
effective room - and elevated-temperature strengthening
agent in these steels. The strengthening mechanism is

associated with the precipitation of a FeMoCr intermetallic compound. On the basis of the work on laboratory-sized heats, a nominally 0.15 C - 14.5 Cr - 5 Mo - 0.5 V - 13.5 Co steel, Steel AFC 77, was selected for scaling-up for production on mill facilities. This steel was produced by air-induction and vacuum-arc-remelting melting methods and processed to different bar and sheet products. No inherent difficulties were encountered in mill-processed products were in good agreement with the properties of the laboratory-sized heats. In general, no significant variation in properties were attributable to the melting method or the product shape or size. As a result of this project, a new hardenable high-strength stainless steel, referred to as Steel AFC 77, has been developed. Steel AFC 77 has very high strength (290,000 psi tensile strength) at the ambient temperature and retains its strength remarkably well up to about 1200°F (120,000 psi tensile strength at 1200°F). In fact, Steel AFC 77 has appreciably higher strength, particularly in prolonged exposures, than any stainless steel known today. The excellent strength properties over a wide range of temperatures are accompanied by good ductility; indications of good formability and weldability have also been obtained. In addition to its superior mechanical properties, Steel AFC 77 is resistant to atmospheric corrosion and oxidation. Therefore, this new steel constitutes a significant advance in the technology of stainless steels and in the technology of elevated-temperature materials.

ASD TR 61-428

March 1962

SUBJECT: AUSFORM PROCESSING OF STEEL BY FORGING
 INVESTIGATOR: R. P. Sernka, R. E. Heise, S. T. Ross
 CONTRACT: AF33(616)-7510, Aeronutronic Division
 ABSTRACT: The strength of some steels has previously been shown to be significantly increased by plastic deformation of the austenite phase while in the metastable condition. This strengthening method has been termed the Ausform process. This investigation has demonstrated the feasibility of Ausform strengthening heavy steel sections utilizing hammer forging as the deformation method. Open and closed-die forgings of Ladish D6-AC steel and open-die forgings of type H-11 steel were processed to several deformations and several tempering temperatures. Mechanical properties and microstructural evaluations were performed on the resulting forgings. Results have shown up to 40 percent increase in yield strength and 35 percent increase in tensile

ASD TR 61-428 (Continued)

strength accompanied by a 4 to 6 point Rockwell hardness increase. Strength and hardness vary directly with amount of Ausform deformation. Strength, hardness, and microstructural evaluations demonstrate that the tempering response of these steels is somewhat retarded after Ausform processing.

HIGH TEMPERATURE METALS

WADC TR 59-575, Part II, OTS Release

June 1961

SUBJECT: OXIDATION OF TUNGSTEN AND TUNGSTEN BASED ALLOYS
INVESTIGATOR: P. E. Blackburn, K. F. Andrew, E. A. Gulbransen, F. A. Brassart
CONTRACT: AF33(616)-5770, Westinghouse Electric Corp.
ABSTRACT: This paper describes the results of studies related to the oxidation of tungsten and its alloys.

The pressure of WO_3 polymers over WO_2 was measured in a tungsten Knudsen cell and found to agree with measurements in a platinum cell. Literature data for WO_2 and WO_3 were combined with vapor pressures determined in this project to give thermodynamic values for $W_{180}49$ and $W_{200}48$.

Tungsten oxidation rates have been measured from 800 to 1700°C and in pressures of oxygen between 2×10^{-1} and 10^{-2} atmospheres. The effects of oxygen pressure indicate that the rate may be governed by oxygen dissociating to atoms at the reacting surface. The oxidation rate is demonstrated to be independent of the oxide evaporation rate. All of the evidence indicates that if an oxide barrier layer is present at temperatures above 800°C it must be very thin.

Studies on the oxidation of tantalum-tungsten alloys between 800 and 1200°C indicate that the 50-50 alloy has the greatest oxidation resistance, oxidizing at a rate as much as 10 times slower than tungsten alone.

WADD TR 60-418, Part II, OTS Release

August 1961

SUBJECT: DEVELOPMENT OF OPTIMUM METHODS FOR THE PRIMARY WORKING OF REFRACTORY METALS
INVESTIGATOR: R. W. Tombaugh, R. O. Green, J. H. Gehrke
CONTRACT: AF33(616)-6377, Harvey Aluminum, Inc.
ABSTRACT: The objective of this program was to provide for the research and development of new processes and techniques for the primary working of refractory metal alloys by hot extrusion. The process developed during the first years' work has been considerably improved and has been used successfully in extruding refractory metals at temperatures above 4000°F. This has required the development

WADD TR 60-418, Part II (Continued)

of improved high temperature billet lubrication, novel die designs utilizing ceramic facing materials, high temperature heating facilities and methods for accurately sensing billet temperatures. This work has been performed at the Metallurgical Experimental Plant of the Aeronautical Systems Division and the operating level of the equipment located therein has been raised accordingly. The suitability of the process has been established by metallurgical evaluation of extruded materials which have demonstrated a high degree of amenability to subsequent reworking by conventional methods. In addition, the extrusion process as developed has been applied to a wide variety of refractory metal alloys and has been useful in providing a substantial quantity of wrought materials for other contractors and governmental agencies.

WADD TR 60-418, Part III, OTS Release

January 1962

SUBJECT: DEVELOPMENT OF OPTIMUM METHODS FOR THE
PRIMARY WORKING OF REFRACTORY METALS
INVESTIGATOR: P. S. Duletsky, V. DePierre
ABSTRACT: Extrusion techniques and processes developed at the experimental extrusion facility, WPAFB, Ohio, were used to provide wrought materials for other contractors and government agencies. These techniques and processes helped to expedite alloy development programs in refractory metals.

Improved lubrication techniques using Corning 7900 glass mixtures were developed for extruding refractory metals at 4000°F.

The suitability of zirconium oxide ceramic coated steel dies for extrusion at 4000°F was confirmed.

Improved extrusion techniques at the experimental extrusion facilities at WPAFB give reproducible, good quality, round and rectangular bar extrusions at temperatures up to 4000°F and reduction ratios of 9.5 to 1.

ASD TR 61-106

May 1961

SUBJECT: INVESTIGATION OF THE PROPERTIES OF
TANTALUM AND ITS ALLOYS
INVESTIGATOR: F. F. Schmidt, W. D. Klopp, D. J. Maykuth,
H. Ogden, R. Jaffee

WADC TR 53-373 Sup 9

15

ASD TR 61-106 (Continued)

CONTRACT: AF33(616)-5668, Battelle Memorial Inst.
ABSTRACT: The effects of alloying on the mechanical properties of tantalum have been studied. Both dispersion-strengthened and solid-solution strengthened tantalum alloys exhibit high-strength at elevated temperatures while maintaining good fabricability and excellent low-temperature ductility. Strength data to 1650°C (3000°F) are reported. The oxidation resistance of tantalum can be improved several fold by alloying. Several alloying elements were found to be effective in reducing both scaling and contamination up to at least 1400°C (2550°F). References are included.

WADD TR 61-123, OTS Release

August 1961

SUBJECT: PROPERTIES OF YTTRIUM AND THE RARE EARTH METALS - OXYGEN AND ALLOY SYSTEMS
INVESTIGATOR: B. Love
CONTRACT: AF33(616)-6829, Research Chemicals Div.
ABSTRACT: Alpha yttrium and erbium are miscible. In the yttrium-neodymium system there is partial solubility and an intermediate phase is present. The solubility of oxygen is low in yttrium, erbium, neodymium, and samarium. Alpha-beta transformations are essentially unaffected. A high temperature monoxide is proposed. The solubility of erbium in cobalt is low. The first compound is Co₁₇Er₂.

Tantalum and niobium form extensive liquid immiscibility regions with rare earths, terminating in a monotectic near the tantalum (niobium) end of the systems; a eutectic at the rare earth end. Solubility is very low. Improved atmospheric corrosion resistance was found for some niobium and cobalt compositions with rare earth additions. The tensile properties of yttrium were improved by alloying with erbium or zirconium. Purification of yttrium and erbium, and improved analytic methods are described. Beryllium with erbium additions shows grain refinement.

ASD TDR 62-7, OTS Release

April 1962

SUBJECT: RESEARCH TO DETERMINE THE COMPOSITION OF DISPERSED PHASES IN REFRACTORY METAL ALLOYS
INVESTIGATOR: F. B. Cuff, Jr.
CONTRACT: AF33(616)-7671, Advanced Metals Research Corp.

WADC TR 53-373 Sup 9

16

ASD TDR 62-7 (Continued)

ABSTRACT: The composition, structures and lattice parameters of the precipitates in two Mo-base and two Nb-base alloys have been determined using the electron micro-beam probe and x-ray and electron diffraction techniques.

In general, the precipitates were found to be complex carbides which, in some cases, could be explained on the bases of known phase relationships and thermodynamic data. The deviations of lattice parameters from those of pure carbides could be explained on the basis of atom substitution.

ASD TDR 62-25, Part I, OTS Release

SUBJECT: INVESTIGATION INTO THE TUNGSTEN-RICH REGIONS OF THE BINARY SYSTEMS TUNGSTEN-CARBON, TUNGSTEN-BORON AND TUNGSTEN-BERYLLIUM PART I. Tungsten-Carbon Equilibrium System

INVESTIGATOR: H. J. Goldschmidt, J. A. Brand

CONTRACT: AF61(052)-306, B.S.A. Research Centre, Birmingham, England

ABSTRACT: A study has been made of the tungsten-rich part of the tungsten-carbon system principally by x-ray diffraction techniques. It has been shown that the solid solubility of carbon in tungsten is finite, attaining a maximum of approximately 0.3 atomic percent at the autectic temperature but decreasing rapidly to 0.05 atomic percent C near 2000°C and to insignificant amounts at lower temperatures. The precipitating phase is W_2C . This has a second allotropic form of cubic (NaCl type) structure, stable at very high temperatures.

PHYSICAL METALLURGY

WADC TR 58-615, Part III

March 1962

SUBJECT: A COMPENDIUM OF CONSTITUTIONAL TERNARY
DIAGRAMS OF THE METALLIC SYSTEMS
ABSTRACT: This is a compilation of constitution
diagrams of ternary alloys of various metallic systems.
In those cases where the ternary diagrams were not complete
or available in the open literature they have been constructed
from the most reliable binary diagrams available. The binary
diagrams used are presented and fully discussed in the first
part of this report. The metallic systems are arranged and
presented in the order of the atomic numbers of the elements
from the lowest to the highest figure to facilitate ease of
locating.

WADD TR 60-37, Part II, OTS Release

May 1961

SUBJECT: PHYSICAL METALLURGY OF TUNGSTEN AND
TUNGSTEN BASE ALLOYS
INVESTIGATOR: H. G. Sell, G. H. Keith, R. C. Koo,
R. H. Schnitzel, R. Coth
CONTRACT: AF33(616)-6933, Westinghouse Lamp Div.
ABSTRACT: Fundamental properties of high purity
tungsten produced both by powder metallurgy techniques and
in the form of single crystals, by electron beam zone melting
were studied. High temperature metallurgical properties of
several dispersed second-phase tungsten base alloys were also
investigated. The mechanism of purification in tungsten
during zone melting was studied. The flow and fracture
characteristics of tungsten single crystal were investigated
and internal friction measurements were carried out at 20-
800°C. Methods of dosing interstitials into tungsten were
explored. The high temperature strength of the alloys
W-ThO₂ and W-TaC was confirmed. A theoretical investigation
of the elastic properties of tungsten and other b.c.c. metals
were initiated.

WADD TR 60-287, OTS Release

March 1961

SUBJECT: REPLICATION TECHNIQUES FOR ELECTRON
MICROSCOPIC STUDIES OF IRON WHISKER
SURFACES
INVESTIGATOR: A. Revere
CONTRACT: AF33(616)-6604, Althea Revere
ABSTRACT: An adaptation of the polystyrene-silica

WADD TR 60-287 (Continued)

technique has been developed for preparing replicas of the surfaces of iron whiskers. By this technique the original whisker specimen is neither sacrificed nor altered. A number of replicas can be made on the same specimen to permit the replication and study of the whole whisker.

Electron micrographs have been taken on replicas of the surfaces of iron whiskers from boats which were prepared by varying the growth conditions. These micrographs show fine details in surface structure which include minute depressions, elevations, and fine parallel grooves and ridges. No interpretation of these structures is made at this time. They will be used in studying growth mechanisms and may facilitate the analysis of the mechanical behavior of iron whiskers at room and elevated temperatures.

WADD TR 60-475, Part I

February 1961

SUBJECT: COLLISIONS OF LIQUID DROPS WITH LIQUIDS
INVESTIGATOR: O. G. Engel
CONTRACT: AF33(616)-59-3, National Bureau of Standards

ABSTRACT: The information available on collisions of liquid drops with liquids is reviewed and some preliminary quantitative data on cavity depth and depth/diameter ratio for collisions of waterdrops with water are given. A plot of the depth of cavity in the target liquid against time elapsed since the collision can be fitted by a parabola or by an ellipse tangent to the cavity-depth axis at the origin. The depth/diameter ratio for the cavity formed in the target liquid is found to vary with time elapsed since the collision; the cavity has a depth/diameter ratio of 0.5 at only two points in time. An equation for maximum cavity depth is derived on the assumption that, at the time of maximum depth, the volume of the cavity in the target liquid is proportional to the kinetic energy of the impinging drop. Apparatus that is now being assembled at the NBS to carry out an exhaustive study of this type of collision is described.

WADD TR 60-864

April 1961

SUBJECT: THE TECHNOLOGY OF SCANDIUM, YTTRIUM AND
THE RARE EARTH METALS - A LITERATURE
SURVEY

WADC TR 53-373 Sup 9

19

WADD TR 60-864 (Continued)

INVESTIGATOR: B. Love
CONTRACT: AF33(616)-6829, Nuclear Corp. of America
ABSTRACT: A survey has been made of the unclassified literature relating to the rare earth elements. The best available data has been compiled for the abundance and distribution of the elements, the methods of recovery from ores, separation and purification techniques, and procedures for reduction to metal. The physical, chemical, and mechanical properties of the elements and their important compounds are presented.

WADD TR 60-894

August 1961

SUBJECT: THE METALLURGY OF SCANDIUM
INVESTIGATOR: D. Geiselman
CONTRACT: AF33(616)-6262, Union Carbide Metals Co.
ABSTRACT: This study of the metallurgy of scandium was undertaken to determine some of the unknown physical electrical, chemical, and mechanical properties. Sublimed calcium-reduced metal was used to study these properties which are more nearly like those of yttrium and the rare earths than those of aluminum. Some mechanical and physical properties of scandium-titanium alloys are given. Limited data on the effect of scandium additions to titanium and chromium are also presented. In all cases the behavior of scandium was very much like that of yttrium.

WADD TR 60-900, OTS Release

September 1961

SUBJECT: CORRELATION BETWEEN VARIOUS PHYSICAL AND CHEMICAL PROPERTIES OF OXIDE SURFACES AND THEIR CONSTITUTION
INVESTIGATOR: T. J. Gray, S. F. Hulbert, D. Benson
CONTRACT: AF33(616)-6188, State University of New York College of Ceramics
ABSTRACT: A study has been inaugurated to elucidate the correlation between physical and chemical properties of oxide and other surfaces and their constitution. A mass spectrometer is under construction to investigate the kinetic aspects of surface properties at high temperature under various incident radiations or under molecular beam impact. Preliminary work using associated equipment concerns the preparation and modification of thin films of oxide by metal evaporation and oxidation techniques during the course of which oxidation characteristics are studied.

WADC TR 53-373 Sup 9

20

January 1961

SUBJECT: RESEARCH ON THE DISTRIBUTION OF TENSION
IN NOTCHED CONSTRUCTION PARTS

INVESTIGATOR: I. H. Neuber

CONTRACT: AF61(514)-1111, Technische Hochschule,
Munchen, Germany

ABSTRACT: The influence of various factors(notch-
radius, depth of notch, notch-angle, material) on the stress
concentration of notched flat bars was investigated theo-
retically and experimentally. For the experimental determi-
nation of the strains and stresses electrical strain gages
were used. Measurements were made by means of the photo-
elastic stress-coat method.

For the elastic range stress-concentra-
tion and stress distribution were evaluated theoretically
and experimentally. For the plastic range, a method was de-
veloped using the real stress-strain curve of the material.
Diagrams for four different materials (steel St. 00.12, St.
00.21, pure aluminum and aluminum alloy "Velodur") were
evaluated in this way, which show the SCF vs the nominal
stress. Numerous tests with notched specimens of the above
mentioned materials confirm the theory; the strain distri-
bution over the minimum cross section was also determined.
Theoretical investigations concerning the treatment of V-
shaped notches and influences of the notch-angle in pure
shear were made.

September 1961

SUBJECT: RESEARCHES ON HYDROGEN OVERVOLTAGE ON
METALLIC SINGLE CRYSTALS: BISMUTH

INVESTIGATOR: L. Peraldo Bicelli, A. La Vecchia, M.
Graziano

CONTRACT: AF61(052)-144, Laboratori Di Electro-
chimica

ABSTRACT: Hydrogen overvoltage on bismuth poly-
crystalline and single crystal cathodes, oriented following
the (100), (110) and (111) planes, has been investigated in
perchloric acid solutions in a c.d. range to 100 A/m² at
25 and 55°C. The Tafel law holds true at high c.d. values;
the parameters being different for the different electrodes.
In the lower c.d. range, the behavior, as revealed by
oscillographic recordings, is anomalous and different from
what observed with other metals.

WADD TR 61-39

SUBJECT: VAPOR PLATING OF ALUMINUM ON COPPER
INVESTIGATOR: E. H. Maus, Sr.
CONTRACT: AF33(616)-7204, Systems Research Laboratories, Inc.

ABSTRACT: Research has been conducted with the objective of developing a new reproducible system for depositing aluminum from a gaseous aluminum compound on copper, steel and magnesium alloy substrates by thermal decomposition. Deposition from the vapors of aluminum acetylacetonate, aluminum iodide and aluminum trimethyl was not successfully accomplished. While certain apparent advantages theoretically favored the use of these compounds, practical processing techniques could not be found. Ductile, adherent, lustrous films of aluminum were successfully deposited on copper substrates by disproportionation of aluminum chloride at 240°C and 4000 microns Hg pressure. Reduction of aluminum chloride anhydrous with hydrogen at 960°C and atmospheric pressure resulted in an AlCu eutectic.

WADD TN 61-54, OTS Release

June 1961

SUBJECT: STUDIED ON IRON WHISKERS OF HIGH AVERAGE STRENGTH GROWN IN A HYDROGEN CONTAMINATED ARGON ATMOSPHERE

INVESTIGATOR: J. E. Emrick

ABSTRACT: The procedures for the growth of iron whiskers from the hydrogen reduction of ferrous chloride in atmospheres of 97% and 0.1% hydrogen are described. The surface conditions and cross-section of these whiskers were determined by light microscopy. These results are presented along with the mechanical properties of iron whiskers as determined by tensile testing at room temperatures. Iron whiskers of high average strength were obtained and tensile tested to establish that studies on high strength whiskers are feasible using techniques which are now fairly routine.

WADD TR 61-58, Part I, OTS Release

April 1961

SUBJECT: THE EFFECT OF SURFACE-ACTIVE AGENTS ON THE MECHANICAL PROPERTIES OF METALS.
PART I. The Effect of Surface Removal on the Plastic Behavior of Aluminum Single Crystals

INVESTIGATOR: I. R. Kramer

CONTRACT: AF33(616)-6220, The Martin Company

WADC TR 53-373 Sup 9

22

WADD TR 61-58, Part I (Continued)

ABSTRACT: Aluminum single crystals were pulled in an electrolytic cell and the surface of the crystal was removed during the deformation. The extent of Stages I and II was increased and the slopes decreased as the metal removed from the surface was increased.

Increasing the strain rate caused a decrease in the extent of Stages I and II and increased the slopes. The experimental data indicate that the work-hardening coefficient of State I is determined primarily by the conditions which exist on the surface of the crystal. In Stages II and III, both surface effects and internal barriers are important.

WADD TR 61-58, Part II, OTS Release

April 1961

SUBJECT: THE EFFECT OF SURFACE-ACTIVE AGENTS ON THE MECHANICAL PROPERTIES OF METALS.
PART II. The Effect of Surface-Active Agents on the Mechanical Behavior of Aluminum Single Crystals

INVESTIGATOR: I. R. Kramer

CONTRACT: AF33(616)-6220, The Martin Company

ABSTRACT: Single crystals of aluminum were pulled in tension in a solution of paraffin oil and stearic acid. The critical resolved shear stress did not change with the concentration of the stearic acid solution; however, the extent and slopes of Stages I and II were affected greatly. The observations lend evidence that the weakening effect of surface-active agents is controlled by the rate of desorption of the metal soap formed by the reaction of the surface-active agent and the metal surface.

ASD TN 61-73, OTS Release

February 1962

SUBJECT: RESEARCHES ON HYDROGEN OVER-VOLTAGE ON METALLIC SINGLE CRYSTALS: ZINC

INVESTIGATOR: P. Bicelli, M. Graziano

CONTRACT: AF61(052)-144, Laboratori di Electrochimica, Milano

ABSTRACT: Hydrogen over-voltage on zinc polycrystalline and single crystal cathode, oriented following the (001) and 1120 planes, has been investigated in 0.01 M perchloric acid and in 0.01 M Hydrochloric acid solutions, in a c.d. range up to 100 A/m², 25 and 40°C.

ASD TN 61-73 (Continued)

The Tafel law holds true at high c.d. values; the parameters being only slightly different for the different electrodes.

WADD TR 61-138

May 1961

SUBJECT: THE EFFECT OF CONCURRENT STRAINING AND A 1-PERCENT MAGNESIUM ADDITION ON THE BEHAVIOR OF ALUMINUM

INVESTIGATOR: T. E. Tietz, C. L. Meyers, J. L. Lytton

CONTRACT: AF33(616)-7156, Lockheed Aircraft Corp.

ABSTRACT: Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with *p*-dibromo- and *p*-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analagous reaction was also studied to determine the possibility of catalysis by ultraviolet light. Convenient procedures were developed for the preparation of *p*-fluoro-, *p*-bromo-, and *p*-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium *p*-bromo-thiophenoxide and having a D.P. of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.

WADD TR 61-145, OTS Release

April 1961

SUBJECT: DISLOCATION AND PLASTIC BEHAVIOR OF IRON SINGLE CRYSTALS

INVESTIGATOR: P. J. Fopiano, S. A. Kulin

CONTRACT: AF33(616)-6348, Manufacturing Labs, Inc.

ABSTRACT: The material in this report is primarily devoted to work carried out during the period 1 February 1960 to 31 January 1961. The factors affecting the growth of iron single crystals by the strain anneal method have been of continuing interest throughout the entire period. The metallography and etch pitting of annealed and strained single crystal specimens have been developed and shown to give consistant results. X-ray and etch pitting techniques showed quantitative agreement. The double crystal spectrometer is felt, however, to be of most interest in this program. Furthermore, the desirability of being able to follow

WADC TR 53-373 Sup 9

24

WADD TR 61-145(Continued)

the effect of strain on the rocking curve of one particular area of an iron single crystal has led to the construction of a tensile apparatus capable of being mounted directly on the double crystal spectrometer. The methods and results of employing this technique are discussed in considerable detail. The analysis of the results based on a dislocation model is presented.

ASD TR 61-147, OTS Release

September 1961

SUBJECT: NORMAL SPECTRAL REFLECTANCE OF ANODIZED ALUMINUM, MAGNESIUM, TITANIUM AND BERYLLIUM
INVESTIGATOR: J. Janssen, R. Torborg, J. Luck, R. Schmidt
CONTRACT: AF33(616)-6191, Honeywell Research Center
ABSTRACT: An integrating hemisphere reflectometer for the measurement of normal spectral reflectance over the range of 0.4 to 22 microns with specimen temperatures from 100 to 1300°F. Reflectance data on 158 specimens of anodized aluminum, magnesium, titanium, and beryllium are presented. Aluminum anodized in sulfuric acid gave a ratio of solar absorptance to infrared emittance of about 0.3 while Al10AT titanium anodized in sodium hydroxide gave a value of $a/\epsilon = 7$. In general, the anodizing process had a greater effect on the reflectance than did alloying elements in the metal. Measurements at elevated temperatures indicated a loss of water from coatings that contained water.

ASD TN 61-157, OTS Release

March 1962

SUBJECT: RESEARCHES ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS: ANTIMONY
INVESTIGATOR: L. Peraldo Bicelli, A. LaVecchia
CONTRACT: AF61(052)-144, Laboratori di Elettrochimica, Milano, Italy
ABSTRACT: Hydrogen overvoltage on antimony polycrystalline and single crystal cathodes, oriented following the (100), (110), and (111) planes, has been investigated in 0.1 M perchloric acid solutions, in a c.d. range up to 100 A/m².

The Tafel law holds true at high c.d. values; the parameters being slightly different for the different electrodes. In the lower c.d. range, oscillographic recordings revealed the behavior is anomalous and different from that observed with other metals.

ASD TR 61-383, OTS Release

February 1962

SUBJECT: GRAIN BOUNDARY SLIDING IN ALUMINUM AND ITS BINARY ALLOYS

ABSTRACT: A study was made of grain boundary sliding in aluminum-magnesium bicrystals to test a mechanism for sliding proposed in previous work. The mechanism related sliding to slip crossing grain boundaries. An investigation was also made of the effect of a second phase existing in various distribution on grain boundary sliding. Al-3% Cu was used for this study. It was found that for tests at similar strain rates very large differences in grain boundary sliding rate resulted from differences in heat treatment. The nature of stresses at the triple points of sliding grain boundaries was also investigated using tricrystals of aluminum and aluminum-magnesium.

ASD TR 61-461, OTS Release

SUBJECT: ELECTRON BEAM WELDING OF TUNGSTEN AND MOLYBDENUM

INVESTIGATOR: H. A. Hokanson, W. I. Kern

CONTRACT: AF33(616)-7439, Hamilton Standard Div.

ABSTRACT: Techniques for electron beam welding of pure tungsten and molybdenum-0.5% titanium were developed and evaluated. Welds were produced in sheet thicknesses to 0.100 in. Primary emphasis was placed on producing welds of maximum strength and ductility. The effects of welding conditions on weld-zone characteristics were evaluated. Butt welds of each thickness exhibiting optimum weld-zone characteristics were evaluated for mechanical properties by bend testing at temperatures to 1200°F. and tensile testing at temperatures to 2800°F. Results of the metallographic and mechanical property investigations of the electron beam welds were compared with base-metal properties and with base-metal properties and with published data for other joining techniques.

ASD TR 61-475, OTS Release

March 1962

SUBJECT: RESEARCH ON HYDROGEN EVOLUTION AND DISSOLUTION

INVESTIGATOR: M. Breiter, L. Kandler, B. Kennel, H. Feigl

CONTRACT: AF61(052)-305, Technische Hochschule, Munich, Germany

ABSTRACT: Investigation of the anion influence on

ASD TR 61-475 (Continued)

the shape of the current potential curves of the ionization of adsorbed hydrogen on platinum. The curves were measured with the potentiostatic method of applying a triangular voltage. Determination of the adsorption isotherms and the differential heat of hydrogen adsorption in dependence upon the coverage and the anions. Study of the change of the surface with time after the anodic activation on platinum. Influence of different additions to inorganic acids on the current potential curves of hydrogen ionization and hydrogen adsorption on platinum.

ASD TR 61-559

February 1962

SUBJECT: POWDER METALLURGY OF COLUMBIUM
INVESTIGATOR: T. L. Robertshaw, M. A. Levinstein,
J. W. Pugh
CONTRACT: AF33(616)-7254, General Electric Co.
ABSTRACT: Powder metallurgy production of four-inch wide, 0.040 inch thick sheet of F-48, a complex columbium base alloy, was the major achievement of this program. The yield of material from billet to sheet was two to three times higher than that experienced by others in processing arc cast material. Two powder metallurgy techniques were successful in the production of billets; vacuum hot-pressing and hydro-pressing and sintering, with hot-pressing achieving a greater degree of development. A third method of consolidation, arc-plasma spraying, proved unfeasible.

The mechanical properties of the hot-pressed sheet product were about 90 percent as strong as the arc cast material and quite as ductile. Despite the fact that the oxygen content was about five times higher than the arc cast material, the sheet was workable; also, preliminary conclusions are it is formable and weldable comparable to arc cast sheet. Only limited evaluation was made of the hydro-pressed sheet product yet mechanical properties were encouragingly good. Low oxygen pre-alloyed powders were produced on a laboratory scale but not evaluated.

ASD TDR 62-229, OTS Release

March 1962

SUBJECT: RESEARCHES ON THE INFLUENCE OF SOME MINOR
CONSTITUENTS OF THE SOLUTION ON HYDROGEN
OVERVOLTAGE OF CU SINGLE CRYSTALS

WADC TR 53-373 Sup 9

27

ASD TDR 62-229 (Continued)

INVESTIGATOR: B. Rivolta, A. Pappagallo
CONTRACT: AF61(052)-144, Laboratori di Electtro-
chimica, Milano, Italy
ABSTRACT: The influence of 10^{-5} M/l of Cd, 10^{-4}
M/l of As, Sb, and In in 0.1 M sulfuric acid on the hydrogen
overvoltage of Cu polycrystalline and single crystal cathodes,
oriented following the (111), (110), and (100) planes, has
been investigated.

In the c.d. range studied, due to the
presence of Cd and In, the Tafel line is broken into two
branches, shifted one from the other, having almost the same
b value.

In the presence of Sb, or As, the over-
voltage vs log c.d. plot is still linear, but for As the
slope value is lower.

MECHANICAL METALLURGY

WADC TR 59-416, Part III

June 1961

SUBJECT: INVESTIGATION OF CREEP BUCKLING OF COLUMNS AND PLATES PART III. Creep Buckling Experiments with Columns of 2024-0 Aluminum Alloy

INVESTIGATOR: R. Papirno, G. Gerard

CONTRACT: AF33(616)-5807, New York University

ABSTRACT: Experimental data for short time buckling and creep buckling of aluminum alloy 2024-0 columns at 500°F were collected. Two slenderness ratios $L'/\rho = 40$ and $L'/\rho = 60$ were tested each with both simulated pinned ends and simulated fixed ends. In addition, compressive short time and compressive creep data for the aluminum alloy materials were collected. A technique, whereby end shortening and central deflection data are autographically recorded both during the period when the creep load was being applied and during creep, was used. It was therefore possible to determine initial imperfections by the Southwell method for the creep buckling experiments. Data presented include initial imperfections as well as central deflection and end shortening data. Experimental relationships between applied stress and failure time are presented as well as an analysis of the central deflection and the end shortening data.

WADC TR 59-416, Part IV, OTS Release

July 1961

SUBJECT: INVESTIGATION OF CREEP BUCKLING OF COLUMNS AND PLATES PART IV. Column Creep Buckling Theory and Correlation with Experiments

INVESTIGATOR: G. Gerard, R. Papirno

CONTRACT: AF33(616)-5807, New York University

ABSTRACT: A creep buckling theory is developed, based upon fundamental concepts of a mechanical equation of state to represent the time dependent behavior at instability of columns and also a time dependent formulation of the governing differential equation and stability criterion. The predictions of this theory as well as those of other classical stability hypotheses are then correlated with recent experimental data on creep buckling of 2024-0 aluminum alloy columns. A simplified approach for prediction of creep buckling is also presented and this is correlated with test data on columns of various aluminum alloys, titanium alloys, and 17-7PH stainless steel. Conclusions are drawn

WADC TR 59-416 (Continued)

as to the predictive value of classical stability approaches and to certain important difficulties in correlating the data which are related to the short time failure behavior of columns at elevated temperatures and which seem to have been overlooked in the past.

WADC TR 59-416, Part V, OTS Release

February 1962

SUBJECT: INVESTIGATION OF CREEP BUCKLING OF COLUMNS AND PLATES. PART V. Theory of Creep Buckling of Perfect Plates and Shells

INVESTIGATOR: G. Gerard

CONTRACT: AF33(616)-5807, New York University

ABSTRACT: A theory for creep buckling of perfect flat plates under axial compression is presented which is based on the use of an equation of state to represent the incremental stresses that arise at buckling. The theoretical results which apply to any arbitrary creep properties are directly equivalent to inelastic buckling solutions where now the tangent and secant moduli are strain rate dependent quantities. Based on these results, a general theory for plates and shells under various types of loading is presented as a logical extension of the compression plate case.

ASD TR 60-161, Part II

June 1961

SUBJECT: EXPERIMENTS ON SLIP DAMPING AT ROUNDED CONTACTS

INVESTIGATOR: L. E. Goodman, C. B. Brown

CONTRACT: AF33(616)-6828, University of Minnesota

ABSTRACT: The apparatus described in Part I of this report has been modified and then has been used to obtain measurements of energy dissipation per cycle at the contact between a flat and spheres of various diameters. The dissipation occurred when the specimens were subject to various constant normal loads and tangential oscillating loads in the range $0.45 < \delta^*/\delta \text{ max} < 1$. The results agree well with the theoretical predictions of Mindlin and Deresiewicz in the range examined. Neither the sphere diameter nor the normal load magnitude appear as essential parameters. The results indicate the necessity of using surface rather than bulk elastic constants in the determination of contact damping. The material employed is AISI 316 stainless steel.

WADD TR 308

August 1961

SUBJECT: QUASI-ORTHOGONAL MODES OF DYNAMICAL SYSTEMS
INVESTIGATOR: L. E. Goodman, Y. C. Das
CONTRACT: AF33(616)-6828, University of Minnesota
ABSTRACT: When energy dissipating devices are inserted at the boundaries the analysis of structural vibration problems becomes difficult. The usual classical methods in such cases yield an infinite set of equations for the coefficients of a modal expansion of the solution. The method developed in this report, gives a finite equation for each coefficient of the modal expansion and thus makes it possible to obtain solutions for structural vibration problems with boundary conditions involving the time derivatives.

The structural vibration problem treated is identical with that of the transient analysis of electrical circuits having distributed capacity and inductance and terminated by a lumped resistance. Certain problems in heat conduction also fall within the scope of the method developed.

WADD TR 60-438, OTS Release

March 1961

SUBJECT: ELEVATED TEMPERATURE DYNAMIC ELASTIC MODULI OF VARIOUS METALLIC MATERIALS
INVESTIGATOR: W. H. Hill, K. D. Shimmin
ABSTRACT: The dynamic elastic moduli of 40 metals and alloys of engineering interest have been determined at room and elevated temperatures. Modulus determinations were based upon a relation between the speed of sound in a material and its elastic modulus. A specimen of the material was excited electrostatically and its resonant frequency determined. Knowing the geometry of the specimen, the dynamic elastic modulus was calculated.

Room temperature comparisons of dynamic with static moduli were made in most instances using material from the same bar.

The results of dynamic elastic modulus determinations are graphically presented.

WADD TR 60-450, OTS Release

February 1961

SUBJECT: CORRELATION OF TENSILE PROPERTIES OF STEEL CASTINGS AND MATERIAL IMPERFECTIONS AS

WADC TR 53-373 Sup 9

31

WADD TR 60-450 (Continued)

DETERMINED BY RADIOGRAPHY
INVESTIGATOR: L. J. Mattek, R. D. Woodward
CONTRACT: AF33(616)-6622, Convair
ABSTRACT: The relationships between tensile properties of 410 stainless-steel castings and imperfections, as determined by radiography, have been investigated. The purpose was to establish confidence in a system of evaluating castings by radiographic inspection. Tensile properties investigated were: tensile yield, tensile ultimate, elongation and modulus of elasticity; imperfections were: gas holes, inclusions and porosity; thicknesses were: 0.1", 0.2", 0.3" and 0.6". Test specimens were heat treated to a 180,000 to 200,000 psi.

Statistical analysis of test results is presented.

Relationships between tensile properties and size or intensity of imperfections are represented in tables and graphs.

WADD TR 60-520, Part I

March 1962

SUBJECT: RESEARCH TO DEVELOP METHODS FOR MEASURING THE PROPERTIES OF PENETRANT FLOW INSPECTION MATERIALS
INVESTIGATOR: R. B. McCauley
CONTRACT: AF33(616)-6420, Ohio State University
ABSTRACT: The first phase of a research program to evaluate the fundamental properties and characteristics of penetrant test materials. The investigation treats various properties of penetrants as well as the wetting phenomena and crack penetration. A standard crack system is proposed.

WADD TR 60-580, Part I, OTS Release

January 1961

SUBJECT: INELASTIC DESIGN OF LOAD CARRYING MEMBERS PART I. Theoretical and Experimental Analysis of Circular Cross-Section Torsion-Tension Members Made of Materials that Creep
INVESTIGATOR: S. Dharmarajan, O. M. Sidebottom
CONTRACT: AF33(616)-5658, University of Illinois
ABSTRACT: This investigation presents a new approach to the problem of multiaxial creep. The theory is based on

WADD TR 60-580, Part I (Continued)

the usual assumptions; namely, the directions of the principal stresses and strains coincide, the Hencky-Mises flow condition is valid, and the material is incompressible. It is proposed that load-deformation relations be derived for a specified time so that the theory is independent of time. For uniaxial state of stress the flow condition was assumed to be the isochronous stress strain diagram obtained from constant stress, tension and compression creep curves. The torsion-tension member was chosen to represent the multi-axial states of stress. Sokolovsky's compressible solution was compared with a closed solution based on the assumption of incompressibility. Experimental data were obtained from nylon and polyethylene members tested in a controlled environment room and 17-7PH stainless steel members at 972°F. Good agreement was found between theory and experiment.

WADD TR 60-580, Part II

January 1961

SUBJECT: INELASTIC DESIGN OF LOAD CARRYING MEMBERS
PART II. The Effect of End Conditions on
the Collapse Load of Columns

INVESTIGATOR: G. Costello, O. Sidebottom, E. Pocs

CONTRACT: AF33(616)-5658, University of Illinois

ABSTRACT: A theory was presented for constructing
the load-deflection relation and for determining the collapse
load of a column having any known end condition. A trial
and error solution was required which used interaction curves
and assumed that the inelastic column assumed the shape of
a sine curve. For time independent inelastic deformation,
constant depth of yielding interaction curves were used.
For time dependent (creep) inelastic deformation arc hyper-
bolic sine interaction curves were used. The experimental
part of the investigation included tests of rectangular
section columns made of 17-7PH stainless steel and tested
at room temperature and at 972°F. Several slenderness ratios
were considered, and the columns had end conditions which
were either fixed, equal and opposite end eccentricities,
or unequal end eccentricities. Good agreement was found be-
tween theory and experiment.

WADD TR 60-583, OTS Release

February 1962

SUBJECT: VIBRATIONS OF ELASTIC SYSTEMS TAKING
ACCOUNT OF ENERGY DISSIPATION IN THE
MATERIAL

INVESTIGATOR: G. S. Pisarenko, A. R. Robinson

CONTRACT: AF33(616)-6828, University of Minnesota

WADD TR 60-583 (Continued)

ABSTRACT: This monograph is devoted to an analytical and experimental investigation of vibrations of non-conservative elastic systems in which the source of energy dissipation is irreversible cyclic straining of the material.

Modern methods of analysis of non-linear vibrating systems are extended to treat problems of the flexural vibrations of long bars of constant and variable cross section, short bars and turbine blades. Torsional vibrations of rods are also considered.

Considerable attention is given to the experimental investigation of energy dissipation in the material. Several apparatuses are described and some of the experimental results presented.

WADD TR 60-752

February 1961

SUBJECT: REDUCTION OF THE ENDURANCE LIMIT AS A RESULT OF STRESS INTERACTION IN FATIGUE
INVESTIGATOR: R. Heller
CONTRACT: AF33(616)-7042, Columbia University
ABSTRACT: This paper presents the results of an investigation of the effects of stress interaction on fatigue life of aircraft structural materials subjected to randomized load spectra. All three materials: 2024 and 7075 aluminum and SAE 4340 steel exhibit fatigue lives shorter than those predicted on the basis of the linear (Miner) damage rule. A quasi-linear rule is proposed with a variable, spectrum dependent, endurance limit producing safe life estimates; the dependence of the endurance limit on the stress spectrum and its resulting design inadequacy is shown. Tests were performed on high speed, programmed, rotating bending fatigue machines of special design.

WADD TR 60-779, OTS Release

March 1961

SUBJECT: VISCOELASTIC SUPPORT JUNCTION DAMPING OF BEAMS
INVESTIGATOR: C. C. Fu, T. J. Mentel, R. L. Schultz
CONTRACT: AF33(616)-6828, University of Minnesota
ABSTRACT: An experimental study, together with evaluation of relevant theory, is conducted of the damping action at the support interfaces of beams which incorporate viscoelastic bonding material. The objective of the study

WADC TR 53-373 Sup 9

34

WADD TR 60-779, OTS Release (Continued)

is the identification of support junction damping techniques which might be of practical use in vibration attenuation. It is found that the highest energy dissipations are associated with flexure and rotation of the beam ends embedded within the support structure.

WADD TR 60-580, Part III, OTS Release

January 1961

SUBJECT: INELASTIC DESIGN OF LOAD CARRYING MEMBERS
PART III. The Significance of an In-
elastic Analysis of Eccentrically-Loaded
Members

INVESTIGATOR: O. M. Sidebottom

CONTRACT: AF33(616)-5658, University of Illinois

ABSTRACT: The author has worked with others on ten investigations, sponsored by Wright Air Development Division which considered the theoretical and experimental inelastic analyses of eccentrically-loaded tension and compression members. In all cases good agreement was found between theory and experiment for members tested at room temperature and at elevated temperatures. This investigation was undertaken to consider the significance of an inelastic analysis of eccentrically-loaded members. If the inelastic deformation can be considered time independent, choice has to be made between an elastic and an inelastic solution. A study was made of the effect of several variables on the ratio of the load necessary to produce a specified inelastic deformation to the maximum elastic load. If the inelastic deformation is time dependent (creep), the only choice is an inelastic solution.

WADD TR 60-580, Part V, OTS Release

January 1962

SUBJECT: INELASTIC DESIGN OF LOAD CARRYING MEMBERS
PART V. Theoretical and Experimental
Creep Analyses of Beam--Columns

INVESTIGATOR: D. L. Dewhirst, O. Sidebottom

CONTRACT: AF33(616)-7600, University of Illinois

ABSTRACT: Theory is presented for constructing load-creep deflection curves for beam-columns at any specified time. The stress-strain-time relation for the material is assumed to be represented by an isochronous stress-strain diagram approximated by an arc hyperbolic sine curve (see Equation 16). A program has been written for the IBM 650 digital computer to calculate points on moment-curvature

WADD TR 60-580, Part V (Continued)

curves for a general I-section. The theory uses these curves and the successive approximations procedure by Newark (32). The experimental part of the investigation included tests of rectangular-and T-section beam columns made of 17-7PH stainless steel and tested at 972°F. Several slenderness ratios were considered. The beam-columns were subjected to a constant axial load located either at the centroid of the section or at an eccentricity of 15 percent of its depth and to a constant transverse load at midspan of sufficient magnitude to produce a linear elastic bending stress when acting alone of $0.50 \sigma_o$.

WADD TR 60-839, OTS Release

March 1961

SUBJECT: THE EFFECT OF CONCURRENT STRESSING ON THE
AIR OXIDATION OF TANTALUM
INVESTIGATOR: B. A. Wilcox, F. H. Beck
ABSTRACT: The air oxidation of unstressed tantalum, measured by decrease in metal thickness over the temperature range 427-649°C (800-1200°F), was found to proceed initially according to a parabolic rate law. At 510-649°C (950-1200°F) oxidation changed from parabolic to linear behavior, after times at temperature sufficient to allow breakdown of the protective initial oxide scale.

Linear and parabolic rate constants were evaluated for the scaling process. Activation energies of 28,600 cal/mole and 40,800 cal/mole were determined for linear and parabolic oxidation, respectively. Oxidation tests in the stressed condition (20,000 and 24,000 psi) increased the parabolic scaling rate about two to four times that of unstressed samples at the same temperature. No enhancement of the linear oxidation rate was noted.

WADD TR 60-854

December 1960

SUBJECT: FATIGUE PROPERTIES OF MAGNESIUM ALLOY
FORGINGS
INVESTIGATOR: E. H. Schuette
CONTRACT: AF33(038)-22609, The Dow Chemical Co.
ABSTRACT: Results are given on axial-load and flexural-bending fatigue tests on specimens from forged-disks of the magnesium alloys AZ31B, AZ71, AZ80A and ZK60A, and for comparison from forged disks of QZ66Xa

WADD TR 60-854 (Continued)

magnesium and 2014 aluminum alloy, rolled rod of 2024, AZ81A, AZ91C, and magnesium sand castings of ZK51A. Results of supplemental tests on full scale I-beams of forged aluminum and magnesium and sand cast magnesium are added. AZ71, AZ80, and ZK60A showed fatigue properties superior to AZ31B. Magnesium forgings compare favorably with aluminum forgings - being superior to magnesium alloy castings - in the absence of sharp notches. Cast magnesium specimens have given results equivalent to specimens cut from forgings. In a production casting, however, it appears that fatigue properties may be impaired by presence of internal stress raisers, which may or may not be visible.

WADD TR 60-920

April 1961

SUBJECT: ULTRASONIC METHODS FOR THE STUDY OF STRESS CYCLING EFFECTS IN METALS
INVESTIGATOR: R. Truell, B. Chick, G. Anderson, C. Elbaum, W. Findley
CONTRACT: AF33(616)-6945, Brown University
ABSTRACT: The measurement of changes of ultrasonic attenuation and velocity during stress cycling are shown together with the accompanying changes in the metallographic character of the surface sample as determined by acetate replicas and optical methods as well as oxide replicas and electron micrographs. The results of these measurements, especially in the very early stages of stress cycling support the ideas that the cycling is accompanied by a dying out of dislocation camping effects, including recovery, as the result of the immobilization of the dislocation oscillation by slip processes together with the increase in scattering caused by the development of defects and strain regions accompanying further slip. Further advances in this area of work will require the study of high purity and single crystal aluminum. The automatic recording of attenuation and velocity changes has been a major part of the effort thus far, and a discussion of this equipment is given.

WADD TR 61-25

January 1961

SUBJECT: CRITERIA FOR COMPARING THE EFFECTIVENESS OF DAMPING TREATMENTS
INVESTIGATOR: D. J. Mead
CONTRACT: AF61(052)-332, University of Southampton
ABSTRACT: In this report, expressions are derived

for the response of simple vibrating systems, from which criteria have been deducted to indicate the effectiveness of a damping treatment in attenuating the response. The criteria include factors by which the treatment increases the mass and stiffness of the system, together with the loss factor increment. The response quantities considered include bending stresses, accelerations, inertia forces and sound transmission associated with simple vibrating plates under harmonic and random excitation. Coincidence sound transmission is also briefly considered. It is shown that whereas the mass and loss factor increase is always advantageous, a stiffness increase in some instances is detrimental. As an example, three different commercial treatments are compared on the basis of some of the criteria. With low treatment weights, the treatment providing the highest loss factor is superior judged by each criterion, but at higher weights according to some criteria a treatment having a lower stiffness, density and loss factor is more effective. The existence of optimum treatment weights for maximum effect upon the response is also shown by some criteria.

WADD TR 61-44

May 1961

SUBJECT: THE EFFECTS OF STRAIN RATE AND HYDROGEN CONTENT OF THE LOW TEMPERATURE DEFORMATION BEHAVIOR OF COLUMBIUM

INVESTIGATOR: B. A. Wilcox, A. W. Bristane, R. F. Klinger

ABSTRACT: The strain rate and temperature dependencies of the low temperature deformation behavior of fine grained arc-melted columbium, 1 part per million hydrogen (1 ppm hydrogen) were evaluated for tensile strain rates of 0.005, 0.10 and 6.0 in/in/min. The effect of hydrogen content (1, 9, and 30 ppm H) on the mechanical behavior was also investigated in the temperature range 25 to -195°C using a tensile strain rate of 0.005/min.

The existence of a hydrogen-dislocation interaction in columbium was confirmed by: (1) calculation of an apparent activation energy for the early stages of low temperature deformation, (2) observation of a hydrogen induced strain aging peak at -50°C, for columbium containing 30 ppm H, and (3) observation of a serrated stress-strain curve at 25°C in coarse grained columbium containing 89 ppm H.

WADD TR 61-53

July 1961

SUBJECT: FATIGUE SENSITIVITY AND RELIABILITY OF MECHANICAL SYSTEMS, ESPECIALLY AIRCRAFT STRUCTURES

INVESTIGATOR: A. M. Freudenthal

CONTRACT: AF 33(616)-6288, Ohio State University Research Foundation

ABSTRACT: The increasing complexity of structural action and operating conditions of modern aircraft and space structures and the rising demand for reliable estimates of the expected operational life of the designed structure as a function of its anticipated mission, which reflects the realization that such structures must be designed for finite life, puts the designer into the frustrating position of having to choose between the conventional engineering design approach based on "safety factors", and the purely statistical reliability approach based on "mean time to failure", knowing that neither approach is really applicable in the design of large, fatigue-sensitive structures.

The present report attempts to develop an integrated design procedure for structures that have to be designed for a combination of "ultimate load" and "fatigue life" failure criteria, based on a quantitative measure of "fatigue sensitivity". The introduction of this measure permits not only a rational classification of structures in terms of their design fatigue sensitivity, but also their reclassification whenever changes in their operational missions produce significant changes in fatigue sensitivity.

ASD TR 61-63, OTS Release

December 1961

SUBJECT: ELASTIC-PLASTIC DEFORMATION OF A SINGLE GROOVED FLAT PLATE UNDER LONGITUDINAL SHEAR

INVESTIGATOR: M. F. Koskinen

CONTRACT: AF18(600)-957, Mass. Inst. of Technology

ABSTRACT: The development of the plastic strain in a V-grooved flat plate under longitudinal shear was followed from the elastic through the partially plastic to the fully plastic condition for a non-strain-hardening material. The region of plastic flow develops monotonically. Adjacent to the zone of deformation in the fully plastic case there is a region where limited plastic deformation has occurred.

The results for the growth of the plastic

ASD TR 61-63 (Continued)

zone were compared with predictions based on the elastic-plastic solution for an infinite plate and the elastic solution for a finite plate. Agreement is good at low stress levels. At high stress levels, a relatively simple empirical equation, satisfying overall equilibrium, is proposed. Predictions based on elasticity theory alone are shown to be seriously in error.

ASD TR 61-64, OTS Release

December 1961

SUBJECT: HIGH-STRAIN TORSIONAL FATIGUE OF SILVER CHLORIDE
INVESTIGATOR: F. A. McClintock, D. S. Groll
CONTRACT: AF18(600)-957, Mass. Institute of Tech.
ABSTRACT: Conditions for determining the motion of dislocation arrays from plasticity solutions are established. The predicted mode of deformation was observed in aluminum and in silver chloride specimens under torsion. Observations of silver chloride under plastic reversed stress showed the slip usually did not reverse on a given plane, that further forward slip could occur on one plane, that considerable disorganization of the crystal developed before fracture, that cracking can occur on planes of high shear stress without appreciable motion normal to the surface, and that structural inhomogeneities reached a scale of the order of .1 mm. Crack initiation and propagation rates were reasonably well correlated with a crack growth theory based on continuum plasticity.

ASD TR 61-97

March 1961

SUBJECT: INFLUENCE FUNCTIONS IN THE THEORY OF FORCED VIBRATIONS OF MEMBRANES
INVESTIGATOR: I. Torbe, D. E. G. Jones
CONTRACT: AF61(052)-332, University of Southampton
ABSTRACT: In this report, a general method is outlined for the calculation of the response of membranes with arbitrary boundaries to arbitrary loadings. It is assumed that, by projecting the area of the given membrane on the surface of an unbounded membrane and then applying the given loading to this projection, the application of a suitable load distribution around the boundary of the projection will enable us to satisfy the boundary conditions appropriate to the given membrane. An attempt to find the

ASD TR 61-97 (Continued)

distribution in question leads to a logarithmically singular integral equation of an unusual type. A few solutions are outlined.

ASD TR 61-128, OTS Release

August 1961

SUBJECT: THERMO-ELASTIC EQUATIONS FOR A SANDWICH PANEL UNDER ARBITRARY TEMPERATURE DISTRIBUTION, TRANSVERSE LOAD AND EDGE COMPRESSION

INVESTIGATOR: I. K. Ebcioğlu

ABSTRACT: The problem of designing sandwich panels for use under an arbitrary temperature distribution, transverse load, and edge compression has been extremely simplified. The core of the sandwich panel is assumed to be orthotropic and the faces may be of different thicknesses and materials. The general differential equations for a sandwich panel are obtained from the principles of mechanics, and corresponding boundary conditions are formulated from the principle of virtual displacements and variational calculus.

A reduction from five differential equations for a sandwich panel to two independent systems of differential equations is accomplished by the suitable transformations of the independent variables.

The final two systems of differential equations are solved simultaneously for an arbitrary temperature distribution, transverse load, and uniform edge compression with simply-supported boundary conditions. The analysis is greatly simplified by the use of the superposition principle. The theoretical solution is applied to a particular temperature distribution and center deflection in the sandwich panel plotted against loading parameter.

WADD TR 61-132

June 1961

SUBJECT: INVESTIGATION OF FATIGUE BEHAVIOR OF CERTAIN ALLOYS IN THE TEMPERATURE RANGE ROOM TEMPERATURE TO -423°F

INVESTIGATOR: R. J. Favor, D. N. Gideon, H. J. Grover, J. E. Hayes, G. M. McClure

CONTRACT: AF33(616)-6888, Battelle Memorial Inst.

ABSTRACT: The fatigue behavior of certain alloys has been investigated in the temperature range room tempera-

WADC TR 53-373 Sup 9

41

ture to -423°F . The alloys evaluated are materials currently used for components in cryogenic missile systems. The results of an initial literature search are presented graphically as S-N curves and as fatigue strength-temperature cross plots. In the experimental program, equipment was designed to test small sheet specimens in fully reversed bending, constant maximum deflection experiments at temperatures down to -423°F . Detailed descriptions of the equipment and specimens are presented. Fatigue data obtained on 14 alloys at room temperature, -110°F , -320°F , and -423°F are presented graphically. The metallurgical histories and chemical analyses are described.

WADD TR 61-165, OTS Release

December 1961

SUBJECT: SIMULTANEOUS HEATING AND LOADING OF TYPE 321 STAINLESS STEEL AND STRAIN AGING OF COLUMBIUM AND TANTALUM
INVESTIGATOR: A. C. Willhelm
CONTRACT: AF33(616)-7057, Southern Research Inst.
ABSTRACT: The experiments were divided into two separate and somewhat unrelated phases of investigation.

In Phase I, a tensile machine supplied with improved methods of strain-rate control was used for determining the tensile properties of 0.060-in. Type 321 annealed stainless steel sheet under conditions of simultaneous heating and loading. The results were compared to predicted results obtained from a graphical method based on a Mechanical Equation of State.

The Phase II experiments were performed on 0.030-in.-thick sheet specimens of annealed columbium and tantalum to determine the effects of strain aging on the tensile properties of the two materials. The specimens were subjected to tensile loading at strain rates of 0.2, 0.05, 0.002, and 0.00008 in./in./ sec. over a range of temperatures from 100° to 2200°F .

The results from Phase I provide a partial confirmation of the concept of a Mechanical Equation of State as shown by the close correlation between the experimental and predicted data. A review is made of the theories related to the Mechanical Equation of State and the results are discussed in terms of these theories.

WADD TR 61-165 (Continued)

In the Phase II work, both columbium and tantalum exhibited strain-aging properties. Strain-aging effects were more pronounced in columbium. Several anomalous side effects were observed and are discussed. The results are discussed in terms of theory, and a brief description is made of a possible deformation mechanism that appears to be consistent both with opposing theories and the experimental results.

WADD TR 61-177

October 1961

SUBJECT: STRUCTURAL SAFETY UNDER CONDITIONS OF
ULTIMATE LOAD FAILURE AND FATIGUE
INVESTIGATOR: A. M. Freudenthal, M. Shinozuka
CONTRACT: AF33(616)-7042, Columbia University
ABSTRACT: The safety of structures subject to
operational loads that cause fatigue damage as well as to
occasional excessive overloads that might produce ultimate
load failure is analyzed. The general relation between
probability of failure and the reliability or the safety
factor is discussed. A new distribution function of fatigue
lives which is compatible with the distribution function
of fatigue damage is derived from an assumed statistical-
mechanical model for fatigue mechanism. It is shown with
the aid of these distribution functions that the probability
of survival of a structure associated only with fatigue is
reduced significantly when subject to the combination of
risks of ultimate and fatigue failures.

ASD TR 61-191 (I)

August 1961

SUBJECT: THEORETICAL FORMABILITY VOLUME I. De-
velopment
INVESTIGATOR: W. W. Wood, R. E. Goforth, R. A. Ford
CONTRACT: AF33(616)-9561, Chance Vought Corp.
ABSTRACT: This two-volume report presents methods
of determining formability analytically for the twelve most
common processes of forming sheet metal. This method is
based on utilization of a material's mechanical properties
to predict formability. The first volume on development
gives the procedure used to arrive at the objective of
predicting formability. First, basic limit equations are
developed relating geometry of the parts to the material
properties. These equations are used to determine the shape
of the limit graphs and to give indices relating formability

WADC TR 53-373 Sup 9

43

ASD TR 61-191 (I) (Continued)

to the material. Then, experimental parts are formed to position the theoretically shaped curves with the aid of the formability indices.

ASD TR 61-194, OTS Release

January 1962

SUBJECT: EFFECTS OF SEVERE THERMAL AND STRESS HISTORIES ON MATERIAL STRENGTH RATE PROCESS THEORY APPROACH

INVESTIGATOR: C. D. Brownfield, D. R. Apodaca

CONTRACT: AF33(616)-5769, Northrop Corp.

ABSTRACT: Methods and design data have been developed for estimating the design strengths of three high temperature alloys under conditions of severe temperature histories. The rate-process based parameter $T(C + \log t)$ was applied with good results to the thermal exposure strength degradation of two high strength stainless steels, AISI Type 301, extra hard temper and PH 15-7 Mo, RH conditions. The same parameter gave indication of applying to the super alloy Rene' 41 within a limited exposure temperature range. Stress applied during thermal exposure was sampled and found to have little effect in promoting degradation of basic material strength, in most cases. In Rene' 41, stress during thermal exposure in air contributed to surface deterioration which caused large reductions in ultimate tensile strength and tensile elongation.

WADD TR 61-200, OTS Release

SUBJECT: EFFECT OF STRESS ON THE CREEP RATE OF HIGH PURITY ALUMINUM IN THE CROSS-SLIP REGION

INVESTIGATOR: N. Jaffe, J. E. Dorn

CONTRACT: AF33(616)-7213, University of California

ABSTRACT: The effect of stress on the creep rate of high purity polycrystalline aluminum in the intermediate temperature range was investigated by conducting creep tests at a series of different stresses and by decreasing the stress abruptly during the course of primary and secondary creep. Three series of decreases in stress tests were conducted: the stress was decreased in primary and secondary regions of creep at 347.5°K and the stress was decreased in primary creep at 327°K . The results demonstrate the effects of structure and temperature on the stress dependence of the

WADC TR 53-373 Sup 9

44

WADD TR 61-200 (Continued)

creep rate. The trends in the creep rate after drop in stress are complicated and show that recovery takes place.

The experimental results are compared with Friedel's theory for cross-slip, and experimentally determined energy for a construction being in good agreement with the theoretical estimate proposed by Stroh.

ASD TR 61-203, OTS Release

August 1961

SUBJECT: THE MECHANICAL PROPERTIES OF TANTALUM WITH SPECIAL REFERENCE TO THE DUCTILE-BRITTLE TRANSITION

INVESTIGATOR: A. A. Adams, A. Iannucci

CONTRACT: AF33(616)-7173, Materials Research Corp.

ABSTRACT: Tensile tests have been made on specimens of commercial tantalum of different grain-sizes, and the variation of lower yield stress σ_y with grain-size $2d$ has been used to assess the effect of temperature (23°C , -78°C and -196°C) and strain-rate ($1.09 \times 10^{-3}/\text{sec}$. and $9.96 \times 10^{-2}/\text{sec}$. at each temperature) on the parameters σ_i and k_y in a Petch type equation $\sigma_y = \sigma_i + k_y \cdot d^{-1/2}$. The results indicate that the sensitivity of the yield strength to temperature and strain-rate arises almost entirely from the effects that these variables produce on σ_i which is in agreement with observations for other body centred-cubic transition metals. The high resistance of tantalum to brittleness has been confirmed, all of the specimens showing pronounced necking and predominantly fibrous fractures; however, on the fracture surfaces of the coarser grained specimens tested at -196°C (both strain-rates) some cleavage facets were observed. This observation of cleavage can be adequately explained by Cottrell's transition equation $(\sigma_i \cdot d^{1/2} + k_y) = \beta \cdot \gamma$ when account is made of the effect of deformation on the parameters in the equation, and a value of the effective surface energy for fracture γ is obtained ($1.35 \times 10^4 \text{ erg. cm}^{-2} \gg \gamma > 1.16 \times 10^4 \text{ erg. cm}^{-2}$) which is in the same range as those values which have been previously measured for niobium, molybdenum and mild steel. The greater ductility of tantalum relative to these materials is attributed to a low value of k_y and a high value of the shear modulus μ . Little or no twinning was observed in the tests, probably because the material was not of a very high purity.

ASD TR 61-217, OTS Release

SUBJECT: INVESTIGATION OF THE EFFECTS OF MAGNETIC
FLUX ON DISLOCATION MOVEMENT AND ALIGNMENT
INVESTIGATOR: A. C. Eckert, Jr., H. W. Newman
CONTRACT: AF33(616)-7116, Allison Division
ABSTRACT: Observations from previous work at
Allison led to the hypothesis that under some conditions,
magnetic flux influences the movement of dislocations within
the grains of a ferromagnetic material. Based on x-ray
diffraction extinction contrast, a technique has been de-
rived from the work on this project that makes it possible
to make a direct observation of dislocations in nickel foil
samples; the dislocation movements can thereby be followed
when the samples are subjected to magnetic flux. An un-
mistakable change in the imperfection structure was observed.
It is recommended that this observation be followed by a
more complete investigation of the nature of the change and
the manner in which it is influenced by magnetic flux.

ASD TR 61-264, OTS Release

November 1961

SUBJECT: PHENOMENOLOGICAL THEORIES OF HYSTERETIC
MATERIAL DAMPING WITH APPLICATION TO
THE VIBRATIONS OF CIRCULAR PLATES
INVESTIGATOR: J. S. Whittier
CONTRACT: AF33(616)-6828, University of Minnesota
ABSTRACT: Prediction of hysteretic specific damping
energy of structural materials under combined dynamic stresses
knowing the damping under uni-axial stresses is discussed.
Consideration is limited to bi-axial states of stress where
 $\sigma_{ij} = S_{ij} \sin \omega t$ with the S_{ij} small enough to preclude
stress history effects. Bounds for bi-axial stress damping
are proposed to be the values predicted by assuming purely
dilatational damping and purely distortional damping.
Available published data, though somewhat inappropriate for
this purpose, show qualitative agreement with the predicted
bounds. New experiments for this report involving vibration
decay tests on mild steel beams and circular plates are dis-
cussed with emphasis on accuracy. Data for beams are used
to predict bounds for plate damping. Plate damping data are
bracketed by these bounds which lends confidence in the
theory for this sort of stress. It is concluded that the
theory will give satisfactory results for isotropic, homo-
geneous materials provided the bi-axially-stressed parts
and the specimens for the uni-axial-stress damping experi-
ments are from the same stock and the uni-axial tests match
the temperature and frequency of the application.

November 1961

SUBJECT: STUDY OF THE EFFECT OF MELTING PRACTICE ON THE FATIGUE BEHAVIOR OF HIGH-STRENGTH STEEL

INVESTIGATOR: H. B. Nudelman, J. P. Sheehan

CONTRACT: AF33(616)-6290, Armour Research Foundation

ABSTRACT: Special carbon-aluminum deoxidation practice provided a significant increase in the fatigue properties of an induction-melted nickel-molybdenum high-strength steel. Prot evaluation of cylindrical R. R. Moore fatigue specimens gave E_p/UTS ratios of 0.500 and 0.555 at ultimate tensile strength levels of 274 and 200 ksi, respectively, which represented an improvement over commercial high-strength steels at similar strength levels. Vacuum arc and vacuum induction remelting of specially deoxidized steel and vacuum arc melting of high purity raw materials resulted in fatigue properties inferior to those associated with special deoxidation practice. Silicon had a harmful effect on fatigue properties if the addition was made prior to the aluminum killing treatment. Nickel-molybdenum steels were relatively unaffected by notching, and good notched fatigue strengths were obtained. The fatigue data did not appear to correlate with melting practice on the basis of inclusion content, and this was more evident for the notched investigation.

February 1962

SUBJECT: THE EFFECT OF INTERSTITIAL ATOM-DISLOCATION INTERACTIONS ON THE DEFORMATION BEHAVIOR OF COLUMBIUM, TANTALUM AND 1020 STEEL

INVESTIGATOR: B. A. Wilcox, R. A. Huggins

ABSTRACT: Yield-point return and dynamic modulus of elasticity were used in a study of strain-aging tendencies of arc-melted Cb, Ta, 1020 steel and hydrogenated Cb. Comparison of activation energies for strain-aging with those for interstitial diffusion revealed that hydrogen was responsible for dislocation locking in Cb, and probably in Ta, in the temperature ranges studied. Dislocation densities (determined by Harper's modification of the Cottrell-Bilby analysis) were compared with values obtained by etch-pitting techniques. To obtain a measure of the degree of dislocation locking in Cb as a function of hydrogen content, the lower yield stress of electron beam melted Cb was measured as a function of grain size and hydrogen content (7, 15, 61, and 78 ppm H).

ASD TR 61-357, OTS Release

December 1961

SUBJECT: CREEP UNDER CONTROLLED STRESS-RATES
INVESTIGATOR: B. Albrecht, D. J. Morecombe, A. M. Freudenthal
CONTRACT: AF33(616)-6112, Columbia University
ABSTRACT: The results of uni-axial creep tests under controlled positive, negative and zero stress-rates on aluminum and zinc are presented and evaluated with the aid of four parameter non-linear and visco-elastic model.

The problem of creep and relaxation of a linear visco-elastic cylinder under internal pressure and axial load is analyzed.

ASD TR 61-385, OTS Release

December 1961

SUBJECT: THE ESTABLISHMENT OF A VACUUM ARC MELTING CAPABILITY AT ASD
INVESTIGATOR: D. R. Carnahan
CONTRACT: AF33(616)-7459, Westinghouse Electric Corp.
ABSTRACT: A consumable vacuum arc furnace has been installed and modified at ASD, Wright-Patterson AFB. The furnace was designed to melt up to six inches in length. Design factors taken into consideration on this furnace were substantial water cooling, good arc visibility, voltage sensitivity stinger-rod rigidity and pumping capabilities.

A total of thirty-one molybdenum - .5% titanium ingots were vacuum arc melted in this furnace to check the furnace capabilities and to try different modes of grain refinement.

Grain refinement of molybdenum - .5% titanium has been obtained.

Other areas of discussion are: hot-topping, hardness, and titanium content and distribution.

ASD TR 61-440, OTS Release

November 1961

SUBJECT: A STUDY OF SUBSTRUCTURE AND CREEP RESISTANCE USING NICKEL WITH PRELIMINARY DATA FOR NIOBIUM
INVESTIGATOR: A. P. Coldren, J. W. Freeman

ASD TR 61-440 (Continued)

CONTRACT: AF33(616)-5466, University of Michigan
ABSTRACT: A correlation obtained between instantaneous creep rate and a quantitative measure of the substructure density at the same time during primary creep of nickel at 1550°F and 3360 psi indicated that the development of substructure was responsible for primary creep. The correlation held for substructures developed at higher stress before testing at 3360 psi. These test conditions were used because sub-structures were developed which could be clearly delineated by etch pitting. Attempts to use lower temperatures failed because the substructures could not be clearly delineated, due either to incomplete polygonization or to precipitation interfering with the decorator effect of impurities. The results did show that the non-homogeneity of substructures requires both high resolution and the ability to average measurements if meaningful quantitative measurements are to be obtained. Attempts to correlate substructures with varied creep-rupture properties at 1100°F from hot rolling failed, due to lack of sufficient polygonization for delineation and to interference with the decorator effect by precipitation. Measurable sub-structures could not be produced for testing at 1100°F by other means due to the interference by recrystallization when the strain rate was reduced sufficiently for polygonization. Extremely limited data for niobium indicated that creep-rupture properties were influenced by rolling conditions even though there was very strain hardening in this material.

ASD TR 61-449

October 1961

SUBJECT: TRANSIENTS IN CERTAIN AUTONOMOUS
MULTIPLE DEGREE OF FREEDOM NONLINEAR
VIBRATING SYSTEMS
INVESTIGATOR: P. Sethna
CONTRACT: AF33(616)-6828, University of Minnesota
ABSTRACT: Oscillations of weakly nonlinear autonomous multiple degree of freedom dynamical systems are studied. Nonlinear effects arising from the potential as well as the kinetic energies of the systems and the systems include elements that produce nonlinear dissipative forces.

ASD TR 61-450

October 1961

SUBJECT: MEASUREMENT OF DISPLACEMENTS IN CONTACT-
STRESS EXPERIMENTS

ASD TR 61-450 (Continued)

INVESTIGATOR: G. E. Bowie
CONTRACT: AF33(616)-6828, University of Minnesota
ABSTRACT: A major part of the instrumentation for contact-stress investigations being carried out at the University of Minnesota has the function of providing displacement measurements in the micro-inch range. This report describes a modified phonograph-type displacement pick-up and an interferometer for calibrating it. In addition, there are described a piezoelectric driving transducer used to the load spheres upon which experiments are performed, a capacitance-machine upon which the apparatus is mounted.

ASD TR 61-451

October 1961

SUBJECT: STRESS REDISTRIBUTION IN NOTCHED SPECIMENS UNDER CYCLIC STRESS
INVESTIGATOR: A. Blatherwick, B. Olson
CONTRACT: AF33(616)-6828, University of Minnesota
ABSTRACT: Most materials exhibit a change in stress-strain relationship when subject to fatigue stresses. In this work, the effect of this change on the stress distribution across the throat of notched plate specimens of mild steel is examined. Using a series of strain gages, the strain distribution across the specimens was determined under dynamic conditions for various number of cycles. Tests of unnotched specimens were used to obtain the cyclic stress-strain properties for corresponding numbers of cycles, and from these data the stress distribution in the notched specimens was determined. Good agreement was obtained between the integral of the stress distribution curve and the total load on the specimen. Tests in which the strain amplitude at the notch root was held constant revealed a decreasing maximum stress with fatigue cycles. In another series of tests, in which the load amplitude was constant, the maximum stress amplitude was observed to decrease with number of fatigue cycles, despite an increasing strain amplitude. In both types of test, the stress concentration factor was observed to decrease with increasing average-stress amplitude, and a further decrease occurs with increasing number of fatigue cycles.

ASD TR 61-467, Part I

September 1961

SUBJECT: INTERFACE DAMPING AT RIVETED JOINTS PART I. Theoretical Analysis

WADC TR 53-373 Sup 9

50

ASD TR 61-467, Part I (Continued)

INVESTIGATOR: D. J. Mead, D. C. G. Eaton
CONTRACT: AF61(052)-332, University of Southampton
ABSTRACT: A theoretical examination is made of the energy dissipated per cycle in a riveted lap joint having a visco-elastic interfacial layer, and being subjected to longitudinal harmonic loading. Simplifying assumptions have been made. The magnitude of this dissipation has been computed for a wide range of joint dimensions, dynamic properties of the layer and rivet stiffnesses. Maximised values of the energy dissipation are obtained together with the optimum joint configuration. Simple design rules have been formulated for maximum interface damping.

ASD TR 61-479, OTS Release

January 1962

SUBJECT: A STUDY OF THE MECHANISMS OF THE TITANIUM-LIQUID OXYGEN EXPLOSIVE REACTION
INVESTIGATOR: J. D. Jackson, P. D. Miller, W. K. Boyd, F. W. Fink
CONTRACT: AF33(616)-7595, Battelle Memorial Inst.
ABSTRACT: A mechanism for the LOX-titanium reaction was proposed during a previous investigation (WADC TR 60-258). It was postulated that the impact of a titanium surface immersed in LOX generates sufficient heat to gasify a pocket of oxygen. In addition, the impact tends to compress the oxygen at the local impact sites. A rapid reaction occurs at the fresh surface formed by the impact. The present investigation has established that a fresh titanium surface, formed by rupture of a tensile specimen, would react in gaseous oxygen under approximately 100 psig pressure at temperatures as low as about -250°F. These results tend to substantiate the proposed mechanism.

Means of eliminating or minimizing the reaction of titanium was ruptured in gaseous oxygen were investigated. The addition of HF as a gas to the oxygen resulted in some inhibition. Argon reduced the reactivity of oxygen gas by dilution. Coating the tensile specimens with fluoride-phosphate or with vapor-deposited aluminum did not affect the reactivity. These same coatings furnished some protection to titanium from reactivity during impact under LOX.

ASD TR 61-623, OTS Release

November 1961

SUBJECT: ANALYTICAL FORMULATION OF DAMPED STRESS-STRAIN RELATIONS BASED ON EXPERIMENTAL DATA WITH APPLICATIONS TO VIBRATING STRUCTURES

INVESTIGATOR: T. J. Mentel, C. F. Fu

CONTRACT: AF33(616)-6828, University of Minnesota

ABSTRACT: A technique is presented for the constructions of stress-strain relations based on experimental, cyclic, damping data. The extension of this technique to the biaxial stress case is then shown followed by an example application involving flexural vibrations of a cantilever.

ASD TR 61-646, OTS Release

November 1961

SUBJECT: DAMPING AND FATIGUE PROPERTIES OF SANDWICH CONFIGURATIONS IN FLEXURE

INVESTIGATOR: L. Keer, B. J. Lazan

CONTRACT: AF33(616)-6828, University of Minnesota

ABSTRACT: A combined theoretical and experimental study is undertaken to develop an analytical approach for predicting the damping of sandwich configurations in flexure. The theory developed analyzes the various contributions to total damping, considering stress distribution and unit damping properties of skin and core, and employs a simple summation process to determine the damping of the composite. To confirm the theory a special test set-up was developed in which sandwich configuration were vibrated as free-free beams utilizing electromagnetic excitation. A series of tests were performed on several types of conventional sandwich beams. Damping predicted by the theory is in good agreement with that measured experimentally.

Fatigue tests were also performed in the specially designed damping machine and S-N curves are presented. Methods of failures and influence of discontinuities are discussed.

ASD TDR 62-256, OTS Release

March 1962

SUBJECT: TRANSIENT VIBRATIONS OF A BEAM WITH NON-LINEAR MATERIAL PROPERTIES

INVESTIGATOR: P. R. Sethna

CONTRACT: AF33(657)-7453, University of Minnesota

ABSTRACT: Free vibrations of a simply supported

ASD TDR 62-256 (Continued)

beam with nonlinear viscoelastic material properties and under symmetrical initial conditions are studied. The method of analysis is based on the "Method of Averaging" for discrete systems. The solutions obtained is asymptotically valid for small nonlinear terms. If the material properties are nonlinear and of the type that produce static hysteresis (metallic materials) then the probable nature of the solution is indicated.

ASD TDR 62-336, OTS Release

March 1962

SUBJECT: AN EXPERIMENTAL INVESTIGATION OF THE
DAMPING PROPERTIES OF AQUAPLAS UNDER
RANDOM AND HARMONIC EXCITATION

ABSTRACT: The linearity of the damping and stiffness of Aquaplas F102B damping treatment is proved by tests on a two-layer plate by comparing dynamic properties measured under harmonic and random conditions. Results are used to predict random stress reductions due to Aquaplas being applied to a simple skin-stringer model vibrating under random forcing. Effect of treatment on mode is fully considered in theory. R.M.S. stress reductions of up to 90% were measured on the model along the rivet lines. Limited agreement between prediction and measurement was found.

JOINING

WADD TR 59-404, Part II

June 1961

SUBJECT: DEVELOPMENT OF PARTIALLY VOLATILE BRAZING FILLER ALLOYS FOR HIGH-TEMPERATURE APPLICATION AND RESISTANCE TO OXIDATION

INVESTIGATOR: N. Bredzs, J. Rudy, H. Schwartzbart

CONTRACT: AF33(616)-6882, Illinois Inst. of Tech.

ABSTRACT: The mechanical properties and the resistance to oxidation of 304 stainless steel joints brazed with the following four experimental filler alloys, containing volatile constituents, have been determined: Alloy A: 61% Ni-39% In, Alloy C: 65% Ni-17% Cr-9 Si, Alloy I: 33% Ni-33% Cr-17% In-17% Ge, Alloy N: 35% Ni-24% Cr-26% In-15% Ge. A special brazing technique was developed for brazing these joints. Miller-Peaslee type specimens brazed by this technique were used for the determination of joint strength at room temperature and elevated temperatures up to 1900°F.

WADD TR 60-917, OTS Release

April 1961

SUBJECT: FUSION WELDING OF BERYLLIUM

INVESTIGATOR: B. M. MacPherson, W. W. Beaver

CONTRACT: AF33(616)-6413, Brush Beryllium Co.

ABSTRACT: A background section describing the accomplishments of the Brush Beryllium Co. related to this fusion welding program is included. The standard conditions used for fusion welding of beryllium in this program are also given. Studies of the effects of post-heat treatment and fixturing on fusion welds are reported, along with limited studies of multiple-pass welding and fillet welding of beryllium. The effects of residual impurities on the weldability of beryllium are discussed in the report sections related to the beryllium filler wire development phase. Also, drawn beryllium welding wire was coated with copper, silver, gold, nickel, iron, cobalt, tin, chromium, zinc, and cadmium; the effects of these coated wires upon the fusion weld properties of beryllium are described.

ASD TR 61-124, OTS Release

August 1961

SUBJECT: EXPLOSIVE WELDING

INVESTIGATOR: V. Philipchuk

CONTRACT: AF33(616)-6797, National Northern Div.

ABSTRACT: Methods were developed for the successful

WADC TR 53-373 Sup 9

54

ASD TR 61-124 (Continued)

welding by the use of explosive forces of 4340 steel to 4340 steel, 6Al-4V Titanium to 6Al-4V Titanium, and Molybdenum to 310 Stainless Steel. No success was achieved with B120 VCA Titanium to B120 VCA Titanium and Molybdenum to Molybdenum.

Test specimens were in sheet or strip form. All welds were lap-type, with one piece of metal directly over or lapped over the other. Successful welds of the above three metal combinations indicate that explosive forces can be utilized for lap weldments of sheet metals when the proper test parameters and techniques have been investigated and developed.

ASD TR 61-313, OTS Release

December 1961

SUBJECT: DEVELOPMENT OF LOW TEMPERATURE BRAZING ALLOYS FOR TITANIUM HONEYCOMB SANDWICH MATERIALS
INVESTIGATOR: N. B. Elsner, E. B. Mack, A. G. Metcalfe, W. C. Troy
CONTRACT: AF33(616)-7249, Solar Aircraft Co.
ABSTRACT: The objective of the program was to develop a process for brazing titanium honeycomb sandwich compatible with its aging cycle (below 1100°F). Also, the brazed joints were to be capable of service up to 850°F.

Initial tests indicated that wetting and flow of the braze alloys were negligible on Ti-6Al-4V at 1050°F; however, by increasing the time at temperature (to approximately one hour or more) good wetting and flow occurred. Further investigation revealed that small additions of titanium to the braze alloys permitted brazing Ti-6Al-4V within 3 to 5 minutes at 1050°F.

Preplaced titanium powder at the joints to be brazed also increased wetting and flow and reacted with the braze alloys to raise the remelt temperature of the joints appreciably

Joints brazed with Au-Sn-Ti showed good resistance to corrosion and high temperature air oxidation; however, strength and ductility were low.

ASD TR 61-584

January 1962

SUBJECT: PHYSICAL METALLURGY OF WELDING TUNGSTEN
AND TUNGSTEN-BASE ALLOYS

INVESTIGATOR: J. W. Clark

CONTRACT: AF33(616)-7484, General Electric Co.

ABSTRACT: The flow and fracture of powder metallurgy and electron beam melted W sheet was studied in both the recrystallized and fusion welded conditions. Effects of impurity level, grain size, annealing temperature, and surface condition were investigated. Both EB and TIG welding were employed. Welds in the powder metallurgy sheet were subject to porosity, those in EB melted W were sound. The ductile-brittle transition temperature of electro-polished and stress-relieved welds were equivalent to that of coarse-grained recrystallized W. The transition temperature varied between 180 and 275°C depending largely on the interstitial content, particularly carbon. Binary and ternary alloys containing Ta, Re, Hf, Zr, B, C, Y, and Th were consolidated by EB melting and evaluated. The most ductile alloy (W-Hf-C) had a transition temperature about 50°C lower than high purity W.

ASD TR 61-592

January 1962

SUBJECT: ALLOY SYSTEMS FOR BRAZING OF COLUMBIUM
AND TUNGSTEN

INVESTIGATOR: W. R. Young

CONTRACT: AF33(616)-7484, General Electric Co.

ABSTRACT: Brazing alloy systems based on Cb, Ta, V, Ti, and Zr were evaluated for melting range, wettability and flow, and metallurgical compatibility with columbium alloys and unalloyed tungsten. Joint shear strength, effect on base alloy transition temperature, and compatibility with protective coatings were determined for selected alloys.

NONDESTRUCTIVE TESTING

WADD TR 61-42

May 1961

SUBJECT: ULTRASONIC METHODS FOR NONDESTRUCTIVE
MEASUREMENT OF RESIDUAL STRESS
INVESTIGATOR: F. Rollins
CONTRACT: AF33(616)-7058, Midwest Research Inst.
ABSTRACT: Shear wave birefringence in metal specimens has been studied as possible method of nondestructively measuring residual stresses. It has been established that the stress-induced birefringence in aluminum and steel varies linearly with the magnitude of an applied stress and does not change appreciably as these metals are deformed plastically. Other sources of birefringence have been studied also. Preferred orientation is a major source especially in rolled metal stock. A pulse echo technique for observing shear wave birefringence is described and the current limitations are discussed.

WADD TR 61-42, Part I, OTS Release

May 1961

SUBJECT: ULTRASONIC METHODS FOR NONDESTRUCTIVE
MEASUREMENT OF RESIDUAL STRESS
INVESTIGATOR: F. Rollins
CONTRACT: AF33(616)-7058, Midwest Research Inst.
ABSTRACT: Shear wave birefringence in metal specimens has been studied as a possible method of nondestructively measuring residual stresses. It has been established that the stress-induced birefringence in aluminum and steel varies linearly with the magnitude of an applied stress and does not change appreciably as these metals are deformed plastically. Other sources of birefringence have been studied also. Preferred orientation is a major source especially in rolled metal stock. A pulse echo technique for observing shear wave birefringence is described and the current limitations are discussed.

WADD TR 61-91, Part I, OTS Release

April 1961

SUBJECT: ULTRASONIC METHODS FOR NONDESTRUCTIVE
EVALUATION OF CERAMIC COATINGS
INVESTIGATOR: W. E. Lawrie
CONTRACT: AF33(616)-6396, Armour Research Foundation
ABSTRACT: This report describes investigations into the use of ultrasonics to detect defects in ceramic-metal

WADD TR 61-91, Part I (Continued)

bonds and to measure the strengths of the bonds. In the techniques investigated ultrasonic frequencies from 30 cps to 35 mc/s have been used and in one method two frequencies are used simultaneously. Low frequency energy (14kc/s) has been successfully used to detect defects by decrement measurements. Low frequencies have also been used in further studies of the intermodulation method used to locate regions of bonds in which defects are present. High frequencies, up to 35 mc/s, have been used with a transmission method and visual images of defects are displayed using a simple charge scanning technique. High frequency energy has also been used in the form of surface waves. (This work is a continuation of investigations reported in WADD TR 60-157.)

ASD TR 61-205, OTS Release

September 1961

SUBJECT: INVESTIGATION OF METHODS FOR DETERMINING
ACTUAL FLAW SIZE IN MATERIALS BY NON-
DESTRUCTIVE ULTRASONIC TECHNIQUES

INVESTIGATOR: L. J. Anderson, G. J. Posakony

CONTRACT: AF33(616)-7279, Automation Industries, inc.

ABSTRACT: The work effort under Contract AF33(616)-
7279 was directed toward the development and evaluation of
techniques for determining actual flaw size, orientation and
geometry in materials by nondestructive ultrasonic means.
This has led toward the development of a practical prototype
breadboard instrument which is capable of presenting an iso-
metric projection of inconsistencies in metal parts. This
development has been made possible through the design of a
unique single oscillating transducer. To obtain the three
dimensional pictures, a special mechanical manipulator and
presentation system were designed and built. The special
mechanical manipulator moves the transducer in a path which
allows complete volumetric scanning of a metal block. The
presentation system will portray an isometric presentation
of the metal block and its inconsistencies. Many different
photographs showing results are included in this report.

COATINGS

WADD TR 60-773

July 1961

SUBJECT: COATINGS FOR THE AEROSPACE ENVIRONMENTS
INVESTIGATOR: R. M. VanVliet
ABSTRACT: This report is the collection of papers presented at the Non-Metallic Materials Laboratory, ASD Conference on Coatings for the Aerospace Environment held in Dayton, Ohio on 9-10 November 1960.

The purpose of the conference was to bring together the principal researchers in the field of coatings for space vehicles to acquaint them with Air Force requirements and programs in this field. It is hoped that this conference will promote effective coordination of coating research programs throughout Government, industrial and academic institutions.

CERAMIC

WADC TR 59-300, Part III, OTS Release

August 1961

SUBJECT: RESEARCH AND DEVELOPMENT SERVICES LEADING TO THE CONTROL OF ELECTRICAL PROPERTIES OF MATERIALS FOR HIGH TEMPERATURE RADOMES
INVESTIGATOR: L. M. Atlas, H. H. Nakamura
CONTRACT: AF33(616)-5929, Armour Research Institute
ABSTRACT: The positive temperature variation of dielectric constant of alumina ceramics can be reduced or reversed by additions of 8-20% of SrTiO_3 . High losses are avoided by the use of a C.P. grade titanate. Mixtures of Al_2O_3 and SrTiO_3 form a pseudo binary system with a eutectic near 1625°C at a composition between 65 and 70% SrTiO_3 . The flexural strength of a 14% SrTiO_3 -86% Al_2O_3 ceramic, and its temperature variation up to 1000°C is about the same as that of a high alumina ceramic. Hot pressing very pure alumina powders results in high density but also introduces contamination from even moderately pure graphite dies.

ASD TR 61-51

July 1961

SUBJECT: HEAT BARRIER COATINGS
INVESTIGATOR: B. A. Macklin, J. C. Withers, E. A. Schatz

WADC TR 53-373 Sup 9

59

ASD TR 61-51 (Continued)

CONTRACT: AF33(616)-7376, American Machine & Foundry Co.

ABSTRACT: Gold has the lowest emittance in air at 800°C (0.03) of any material but interdiffuses readily with metallic base materials. A qualitative theory was developed to explain the interdiffusion and allow a prediction of materials to be used as diffusion barrier coatings for gold. Satisfactory diffusion barrier materials investigated were NBS ceramic Al₂O₃, NiO, CeO₂, SiO₂, and a variety of phosphate bonded oxides. NiO and CeO₂ were the best diffusion barrier coatings showing no significant increase in emittance of the gold coating for 110 hours at 800°C. Palladium and rhodium were inferior in many respects to gold as a low emittance high temperature coating.

ORGANIC

WADD TR 61-65

April 1961

SUBJECT: DIELECTRIC MOISTURE BARRIER COATING MATERIALS

INVESTIGATOR: L. M. Kindley

CONTRACT: AF33(616)-7010, Melpar, Inc.

ABSTRACT: The effect of treating the surfaces of filler or pigment particles on the water vapor permeability, water absorption, and adhesion between such surfaces and selected organic vehicles was determined in this study. In addition, the effect of surface treatments on specific mechanical and electrical properties of coating under ordinary and hyperenvironmental conditions was investigated. Coatings formulated from three different resins and five fillers were considered.

A significant increase in the tensile strength of coatings containing treated filler was observed.

There was also an improvement in the dispersion of fillers in organic binders as a result of treating the filler with an organo-silane. The films cast from such formulations were noticeably smoother and more uniform.

Water vapor permeability, water absorption, and other physical properties were not significantly affected by treating fillers before incorporation in the resin and preparation of films.

May 1961

SUBJECT: SYNTHESIS OF METAL CYCLOPENTADIENYL DERIVATIVES FOR USE AS ULTRAVIOLET ABSORBERS

INVESTIGATOR: J. J. Mattice

CONTRACT: AF33(616)-7214, Wyandotte Chemical Corp.

ABSTRACT: In a search for ferrocene derivatives that may be useful as protective ultraviolet absorbers, twelve candidate ferrocenes were synthesized, the spectral characteristics in the 2000 to 5000A region were obtained, and the melting points, micro boiling points, and solubility properties of the ferrocenes were determined. Thus, 2-methoxy-benzoylferrocene, 2,4-dimethoxybenzoylferrocene, benzoylferrocene, 1,1'-dibenzoylferrocene, and diferrocenyl ketone were prepared by Friedel-Crafts reactions on ferrocene. The methoxy compounds were converted to 2-hydroxybenzoylferrocene, 2,4-dihydroxybenzoylferrocene, and 2-hydroxy-4-methoxybenzoylferrocene by treatment with aluminum chloride. Ethyl ferrocenemonocarboxylate and diethyl 1,1'-ferrocenedicarboxylate were prepared from the crude acids, and basic hydrolyses of these esters furnished pure ferrocenemonocarboxylic acid and 1-carboxy-1'-carbethoxyferrocene. At 345°C, ferrocenemonocarboxylic acid decomposed to pure ferrous ferrocenemonocarboxylate. Studies have been continued of the preparation of substituted ferrocenes, particularly those containing oxygenated and nitrogenated groups. The 1,1'-dimethylferrocenedicarboxylic acids have been prepared and partially separated. Nitroferrocene, 1,1'-dinitroferrocene, azoferrocene, azoxyferrocene and aminoferrocene have been prepared and their properties determined.

ASD TR 61-151, Part I

December 1961

SUBJECT: REFLECTIVE COATINGS ON POLYMERIC SUBSTRATES

INVESTIGATOR: R.B. Belser, M. D. Carithers

CONTRACT: AF33(616)-6980, Engineering Experiment Station, Georgia Inst. of Technology

ABSTRACT: The purpose of this research is to determine methods of controlling the optical properties of metal surfaces by over-coating the surface with a plastic film to obtain the proper gloss and a metal film to obtain the desired reflectivity over the wavelength range 1 to 15 microns.

Over 750 specimens of steel or stainless steel have been coated with plastic formulae of the species epoxy, polyurethane, or silicone and subsequently overcoated

ASD TR 61-161, Part I (Continued)

with silver, gold, copper, or aluminum. Adherences, reflectances and emissivities of the various coated surfaces have been measured as well as survival of the plastic films in vacuo at temperatures up to 200°C.

Adherence of the plastic to the base metal was generally greater than 2000 psi while that of the metal films to the plastics was in the range 300 to 900 psi. Reflectances in the range 0.90 to 0.99 over the range 1 to 15 microns were measured for selected films of each metal on each plastic type. Metal film thickness of 2000 to 4000 Angstroms were sufficient for maximum reflectance. Emissivities of the better coated specimens were in the range 0.03 to 0.06 at 100°C.

ASD TR 61-298

July 1961

SUBJECT: STUDIES ON THE PROTECTIVE ULTRAVIOLET ABSORBERS IN A SPACE ENVIRONMENT III.
INVESTIGATOR: R. G. Schmitt, R. C. Hirt
CONTRACT: AF33(616)-5945, American Cyanamid Co.
ABSTRACT: The effectiveness of the ultraviolet absorbers in protecting organic coatings exposed to an extra-terrestrial environment from photochemical degradation was studied for the commercially available absorbers and certain ferrocene derivatives. The change in solar absorptivity of the coatings was used as a measure of the degradation incurred; these data being obtained from the ultraviolet, visible and near-infrared absorption spectra of the polymers. Ultraviolet absorbers of the benzophenone and benzotriazole type used for terrestrial applications were found to be considerably less effective in a space environment. This was attributed to the strong ultraviolet absorption of the polymers at the shorter wavelengths which competes with the absorber for the incident radiation. Certain benzoyl substituted ferrocene compounds were found to be extremely effective in protecting many types of polymeric coatings. Improvement in the stability of the coatings by factors of 30 to 5000 were obtained. The benzoylferrocenes are highly colored materials (red to orange) whose absorbing properties are extremely stable to ultraviolet radiation.

REFRACTORY METAL

ASD TR 60-486, OTS Release

September 1961

SUBJECT: THE EFFECT OF CADMIUM PLATING ON AIR-CRAFT STEELS UNDER STRESS CONCENTRATION AT ELEVATED TEMPERATURES

INVESTIGATOR: E. M. Kennedy, Jr.

ABSTRACT: A study has been conducted of the effects of cadmium plating on stressed steels at elevated temperatures. The experimental procedures involved several tests characterized as the stress-rupture tests, tensile tests, and fatigue tests. Materials studied consisted of several aircraft quality SAE steels: namely 4340, 4130, 1095, 18-8, and H-13 hot work die steels. Strength levels from 180,000 psi to 300,000 psi, as suitable for the several steels, were evaluated for a variety of conditions of stress concentration.

The steels examined, except the austenitic stainless steels, were susceptible to embrittlement by cadmium plating at elevated temperatures. With decreasing temperatures, the noticeable effect of cadmium plating on the properties of steels is correspondingly decreased. All the steels examined, showing an effect on one property showed similar effects on the other properties.

ASD TR 61-66, Part I

May 1961

SUBJECT: DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE PROTECTIVE COATINGS FOR COLUMBIUM ALLOYS PART I. Coating Development

INVESTIGATOR: R. A. Jefferys, J. D. Gadd

CONTRACT: AF33(616)-7215, Thompson Ramo Wooldridge

ABSTRACT: A detailed study was made of the formation and protective nature of surface alloy diffusion coatings for columbium. Vacuum vapor deposited, diffusion alloy coatings combining chromium, titanium and silicon were found to protect D-31 alloy, F-48 alloy and unalloyed columbium from surface oxidation or internal contamination for considerable lengths of time in air in the range of 2000 to 2600°F and for shorter periods up to 2800°F. Tests on the coating-base metal systems included cyclic oxidation, thermal shock and high velocity-hot gas erosion in a plasma flame.

June 1961

SUBJECT: DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE PROTECTIVE COATINGS FOR COLUMBIUM ALLOYS PART II. Coating Evaluation

INVESTIGATOR: R. A. Jefferys, J. D. Gadd

CONTRACT: AF33(616)-7215, Thompson Ramo Wooldridge

ABSTRACT: A comparative evaluation was made of 18 coating-base metal systems, six different coatings applied to 3 columbium base materials (D-31 alloy, F-48 alloy and unalloyed columbium). The 18 coating-base metal systems were tested under the same conditions in cyclic oxidation (2300 and 2500°F) thermal shock (2500 to 2500°F), bend-oxidation (2500°F) and stress-oxidation (2500°F) plus tensile tests. The tests produced directly comparable data between the coating-base metal systems relating to the protective nature of each coating and the effect of the coating and the coating treatment on the mechanical properties of the substrate.

WADD TR 61-137

May 1961

SUBJECT: ALUMINUMPHOSPHATE COATINGS

INVESTIGATOR: E. Ott, E. R. Allen

ABSTRACT: For the protection of metals at higher temperatures and also for modification of surfaces to satisfy varying space requirements, new protective coatings and vehicles are needed. It was believed that some forms of aluminum phosphate might prove useful. The literature is somewhat confusing and the preparation of reliable samples requires considerable research. Aluminum orthophosphate has been prepared in the quartz, tridymite and cristobalite structures. Once formed tridymite and cristobalite, and high temperature forms, persist at room temperature. It may well be possible to obtain these forms stable enough to resist further transitions with their abrupt volume changes. This would greatly increase the potential of $AlPO_4$ as a refractory material.

Perhaps our most important finding is a certain aluminumphosphate with Al/PO_4 ratio of 1.20 which is a crystalline material that sinters or fuses at 875°C to an amorphous glass-like coating. It shows good adhesion to steel and stainless steel. The preparation has been successfully repeated. The product appears promising as a relatively low temperature frit for novel coatings.

October 1961

SUBJECT: HIGH TEMPERATURE OXIDATION RESISTANT
COATINGS FOR TANTALUM BASE ALLOYS

INVESTIGATOR: D. D. Lawthers, L. Sama

CONTRACT: AF33(616)-7462, Sylcor Division, Sylvania
Electric Products

ABSTRACT: Aluminide and beryllide coatings were investigated for pure tantalum, a commercial Ta-10W alloy, and a ternary alloy under development. Coatings were applied by dipping, packing, cold spraying, and vapor deposition. Oxidation resistance was evaluated for furnace testing, resistance heating, and flame testing. Internal hardening and diffusion effects were also studied. A Sn-Al coating was developed which has excellent oxidation resistance for 10 hours to at least 3000°F. Evaluation was successfully extended to arc-plasma tests which also showed the feasibility of protecting molybdenum and tungsten. A similar but minor effort investigation was performed with a columbium alloy which could be protected to 2500°F for at least 10 hours.

February 1962

SUBJECT: SYNTHESIS OF PYROLYSIS OF REFRACTORY
METAL ALKOXIDES

INVESTIGATOR: K. S. Mazdiasni, C. R. Conners, C. A.
Pratt

ABSTRACT: Metal alkoxides were prepared based on the D. C. Bradley, et al procedure. Sufficient quantities of principally zirconium isopropoxide and tetra-tert butoxide were produced to use as starting material for impregnation and coating purposes. The compounds were prepared by simple chemical reactions but under extremely dry atmosphere.

The purity, concentration, order of stability, melting temperature, vapor pressure, and decomposition temperature of the compound are described.

While the compound may be applicable to coating various materials, to date experiments have been limited to coating graphite substrates. The compound was pyrolyzed at 250° - 500°C. Tetragonal-monoclinic thin film ZrO₂ was deposited on graphites of varying purity.

CORROSION

WADD TR 60-179, OTS Release

January 1961

SUBJECT: EVALUATION OF STRESSED AND UNSTRESSED MATERIALS IN SIMULATED HIGH ENERGY FUEL EXHAUST PRODUCT ENVIRONMENTS

INVESTIGATOR: J. W. Rosenberty

CONTRACT: AF33(616)-6198, University of Dayton

ABSTRACT: The effects of corrosion of selected stressed and unstressed high temperature materials by molten boric oxide are presented. A sequence of eleven experimental exposures was accomplished. The effects of small additions of elemental boron and carbon to the molten bath are described. Results show that stress has no significant effect upon the onset or rate of the corrosion process. This report is the last of a sequence of three describing the experimental evaluation of the corrosion of "superalloy" type materials and selected ceramic coatings by boric oxide at temperatures from 1600°F to 2100°F. References: WADC TR 58-443 and WADC TR 59-205.

WADD TR 60-819

March 1961

SUBJECT: THE COMPATIBILITY OF VARIOUS METALS IN LIQUID FLUORINE

INVESTIGATOR: C. J. Sterner, A.H. Singleton

CONTRACT: AF33(616)-6515, Air Products, Inc.

ABSTRACT: Investigation of the compatibility and resistance to corrosion of various alloys of aluminum, stainless steel including high strength steel, titanium, copper, nickel, and magnesium. Tests were designed to investigate the following: change in liquid fluorine-metal corrosion rate with time; the effect of contaminants in liquid fluorine; probability of metal ignitions in liquid fluorine following explosive shock or compressive impact; the change of mechanical properties of metals due to one year exposure to liquid fluorine. The results indicate that liquid fluorine, pure and contaminated with water, is unreactive with metals; fluoride films are unimportant in corrosion resistance to liquid fluorine; impact ignition sensitivity of titanium in liquid fluorine is similar to titanium in LOX.

WADD TR 61-54

April 1961

SUBJECT: THE COMPATIBILITY OF MATERIALS WITH
CHLORINE TRIFLUORIDE, PERCHLORYL FLUORIDE
AND MIXTURES OF THESE

INVESTIGATOR: J. C. Grigger, H. C. Miller

CONTRACT: AF33(616)-6796, Pennsalt Chemicals Corp.

ABSTRACT: Compatibility and corrosion rates of
alloys of aluminum, copper, magnesium, nickel, titanium,
steel and stainless steel, and columbium, molybdenum, carbon,
graphite and fluorocarbon plastics in chlorine trifluoride,
perchloryl fluoride and mixtures of these at 30°C were in-
vestigated. Titanium, columbium, molybdenum, carbon and
graphite were rapidly attacked in ClF₃. Corrosion rates of
others were extremely low in all liquids. In the vapors,
instances of higher corrosion rates were noted. Teflon and
Kel-F adsorbed moderate amounts of ClF₃ and ClO₃F. Passi-
vation by ClF₃ was unnecessary for reducing corrosion of
properly cleaned metals. Corrosion in wet ClO₃F was character-
ized by localized attack, but some stainless steels were re-
sistant. Titanium exhibited increasing impact ignition in
liquid ClO₃F beginning at 19 ft.-lbs., but even at 140 ft.-
lbs. burning was not sustained. No other metals showed im-
pact ignition in ClF₃ or ClO₃F. In explosive shock tests,
ClO₃F gave a stronger interaction with the metals tested
than did ClF₃ and aluminum showed a greater interaction with
the fluorine chemicals than low carbon or stainless steel.
Greatest enhancement of explosive shock occurred with
titanium and ClO₃F. In explosive denting and perforation
of steel and aluminum cylinders containing ClF₃, ClO₃F and
their mixtures, no enhancement occurred. A high order ex-
plosive interaction occurred between ClO₃F and titanium
cylinders perforated by a shaped explosive charge.

ASD TR 61-121

July 1961

SUBJECT: EFFECT OF CORROSION ON THE FATIGUE BE-
HAVIOR OF 2024-T4 ALUMINUM ALLOY

INVESTIGATOR: C. L. Harmsworth

ABSTRACT: An investigation was conducted to de-
termine the effect of corrosion pitting on the fatigue be-
havior of 2024-T4 aluminum alloy and to establish a method
of 2424-T4 aluminum alloy and to establish a method of
measuring pitting corrosion damage with respect to fatigue.
It was found that surface roughness measurements could be
used to give a useful indication of the fatigue life that
may be expected from a corroded structural member of 2024-T4
aluminum. The effect of pre-existing corrosion on the

ASD TR 61-121 (Continued)

mechanism of fatigue was determined to be largely that of a stress raiser. In the case of pitting corrosion, calculations could be made, based upon critical pit measurements, to determine the stress concentration effect of the corrosion pit.

ASD TR 61-175

May 1961

SUBJECT: IMPACT SENSITIVITY OF METALS (TITANIUM)
EXPOSED TO LIQUID NITROGEN TETROXIDE
INVESTIGATOR: H. F. Scott, C. W. Alley, H. T. Gerry,
A. W. Hayford
CONTRACT: AF33(616)-7114, Allied Chemical Corp.
ABSTRACT: The purpose of this investigation was to determine the impact sensitivity of commercially pure titanium, titanium alloy 6Al-4V, and precipitation hardened 15-7 Mo stainless steel when exposed to liquid nitrogen tetroxide. An explanation is given of the probable mechanism of the limited ignition resulting from impact. Commercially pure titanium and titanium alloy 6Al-4V ignite about 50% of the time when impacted with a 0.5 inch diameter flat striker having an energy of 200 ft-lbs. Storage of the commercially pure titanium in N_2O_4 decreases the energy needed for 50% ignition to 60-70 ft-lbs. The sensitivity of the alloy is unaffected by storage in N_2O_4 . Precipitation hardened 15-7 Mo stainless steel was not impact sensitive in liquid N_2O_4 .

ASD TR 61-345

June 1961

SUBJECT: EFFECT OF JET FUEL ADDITIVES ON FILTER-
ABILITY AND WATER SEPARATION CHARACTERI-
STICS
INVESTIGATOR: R. K. Johnston, J. P. Cuellar, Jr.
CONTRACT: AF33(600)-39425, Southwest Research Inst.
ABSTRACT: The effect of corrosion inhibitors on filtration and water separation characteristics of JP-4 fuel was studied in a single-element filter-separator test facility, using five types of commercial elements and housings designed to simulate full-scale flow conditions. Standard coarse A/C dust iron oxides. Adverse effects of the corrosion inhibitors varied widely, and the element types differed in efficiency and modes of failure. The CRC water separator, a bench-scale apparatus, gave a general correlation with the single-element results. Interaction of

ASD TR 61-345 (Continued)

additives with fuel constituents caused difficulty in early work, but otherwise the separometer repeatability was satisfactory. Preliminary work on a reference fluid indicated that an "odorless solvent" may be suitable.

ASD TR 61-676

January 1962

SUBJECT: DEVELOPMENT OF PROTECTIVE COATINGS FOR
TANTALUM-BASE ALLOYS
INVESTIGATOR: W. Klopp, C. Powell, D. Maykuth, H.
Ogden
CONTRACT: AF33(616)-7184, Battelle Memorial Inst.
ABSTRACT: This program was started with a survey
to determine potential coating materials for protecting
tantalum alloys from oxidation and contamination at high
temperatures. This survey and initial coating studies re-
sulted in major emphasis being directed toward silicide-
base coatings. Straight silicide coatings and modifications
with aluminum, boron, chromium, manganese, molybdenum,
titanium, and vanadium were applied to tantalum, Ta-10W,
Ta-10Hf-5W, and Ta-30Cb-5V by pack-cementation techniques.
These coated materials were evaluated primarily by static,
cyclic, and defect oxidation studies in the temperature range
1200 to 2700°F. Less extensive studies were conducted on
tantalum coated with aluminum, chromium, hafnium, titanium,
zinc, and Al₂O₃.

POLYMERS AND SYNTHESIS STUDIES

WADC TR 53-426, Part IX

August 1961

SUBJECT: ORGANO-METALLIC AND ORGANO-METALLOIDAL
 HIGH-TEMPERATURE LUBRICANTS AND RELATED
 MATERIALS

INVESTIGATOR: H. Gilman, W. H. Atwell, F. K. Cartledge,
 O. L. Marrs, G. L. Schwebke

CONTRACT: AF33(616)-6463, Iowa State University of
 Science and Technology

ABSTRACT: A series of new syntheses has been de-
 veloped for the preparation of so-called custom-made molecules.
 Also, some older syntheses have been improved from the points
 of view of better yields and more convenient processes.
 These reactions have been applied to the preparation of organo-
 silicon and related compounds in studies concerned with high
 temperature fluids and lubricants, as well as anti-oxidants
 for high temperature lubricants.

WADC TR 55-58, Part VII

March 1962

SUBJECT: NUCLEAR RADIATION RESISTANT POLYMERS AND
 POLYMERIC COMPOUNDS

INVESTIGATOR: J. W. Born

CONTRACT: AF33(616)-7491, B. F. Goodrich Co.

ABSTRACT: This research includes basic studies of
 radiation energy transfer and mechanisms of radiation damage
 in high polymers; the selection, design, and synthesis of
 special new monomers to produce new high polymers having
 outstanding inherent radiation resistance and heat stability;
 polymerization and copolymerization of said monomers, identi-
 fication of resulting polymers, and evaluation of their heat
 and radiation-induced compression set of various rubber com-
 pounds as a function of cure and of elastomer, antirad,
 plasticizer, aromatic extender, and degree of compression;
 and static and semidynamic radiation testing of "O"-rings
 with attempts to protect the military-approved rubber com-
 pounds against radiation damage.

WADD TR 59-64, Part III

November 1961

SUBJECT: THERMAL DEGRADATION STUDIES OF POLYMERS
 AT HIGH TEMPERATURES

INVESTIGATOR: S. L. Madorsky, S. Strauss, M. E. Wacks

CONTRACT: AF33(616)-58-8 National Bureau of Standards

WADC TR 53-373 Sup 9

70

WADD TR 59-64, Part III (Continued)

ABSTRACT: Section A. Polydivinylbenzene and copolymers of styrene with divinylbenzene and with trivinylbenzene were pyrolyzed in a vacuum in the temperature range 246° to 450°C. The results indicate a gradual increase in thermal stability of the copolymers as the amount of cross-linking agents divinylbenzene and trivinylbenzene is increased. Thermal degradation studies of polystyrene, polymethylene, polytetrafluoroethylene, poly- α -methylstyrene, polypropylene, polyisobutylene and poly(methylmethacrylate) showed that the higher the temperature and pressure of pyrolysis the greater is the fragmentation of the degradation products.

Section B. Instrumentation for the study of the basic properties occurring in the thermal degradation of organic polymers has been investigated. This instrumentation will be used to determine the initial stages of degradation of polymers.

WADC TR 59-272, Part III

August 1961

SUBJECT: SYNTHESIS OF SEMI-INORGANIC FLUORINE POLYMERS
INVESTIGATOR: H. C. Brown
CONTRACT: AF33(616)-6887, University of Florida
ABSTRACT: The formation of an intermediate polymer from the reaction of perfluoroglutaronitrile with perfluorobutyramidine has been studied both in solution and in bulk. This intermediate product, by deammonation under further heat treatment, has been shown to produce the perfluoroalkyl-triazine polymers in thin, coherent sheets that are elastic and thermally stable. A study of the volatile products produced under controlled heating of the intermediate polymer has been initiated.

N¹(perfluoroacylimino)perfluoroalkylamidines, the initial intermediates in the deammonation of perfluoroalkylamidines to tris(perfluoroalkyl) sym. triazines, have been studied in more detail as a part of the overall mechanism of triazine formation. Revised preparative procedures, visible and ultraviolet spectra, analytical procedures and hydrolysis products are described. Deuteration of N-H bonds and spectral shifts are discussed.

A quantitative study of the catalytic

WADC TR 59-272, Part III (Continued)

trimerization of perfluoroacetonitrile and polymerization of perfluoroglutaronitrile is presented. The cotrimerization of perfluoroacetonitrile with organic aromatic nitriles is shown to be much more efficient with the basic catalyst ammonia than with acidic catalysts.

WADD TR 59-345, Part III

November 1961

SUBJECT: HIGH-TEMPERATURE SYNTHESIS OF NEW,
THERMALLY-STABLE CHEMICAL COMPOUNDS
INVESTIGATOR: L. Bratt, D. Chamberlain, T. Mill,
C. Marynowski
CONTRACT: AF33(616)-7245, Stanford Research Inst.
ABSTRACT: Continued studies of energetically
activated synthesis methods for new, thermally-stable com-
pounds and polymers are described. Concurrent investigations
were conducted in the following areas. (1) Synthesis of new
phosphonitrilic compounds by means of electronically acti-
vated reactions or pyrolytic routes, (2) Synthesis of di-
fluoroacetylene and related perfluorocarbon compounds (3)
Synthesis of new fluoro-organic compounds and polymers by
means of plasma-jet pyrolysis of simple halofluoro-carbons.
No useful phosphonitrilic products (both thermally and hydro-
lytically stable) were isolated from any of the electroni-
cally activated reactions. Pyrolytic routes to phosphonitrilics
yielded stable product fractions, one of which had the approxi-
mate empirical formula $(SP_2N_2H_2)_n$; the reactions were difficult
to stop reproducibly at the theoretical product. Attempted
synthesis routes to difluoroacetylene involved either pyro-
lytic degradation or halogen displacement of suitable pre-
cursors; in all cases, either there was no reaction or there
was extensive decomposition without evidence of the formation
of difluoroacetylene. In one reaction (pyrolysis of acetylene
dicarboxyl fluoride), the volatile products condensed to a
stable polymeric film; however, the film was not perfluori-
nated. The plasma jet pyrolysis technique was found to give
only volatile fluorine compounds when the halofluorocarbon
was passed through the plasma. However, evidence was ob-
tained that the quench medium could be disproportionated to
high molecular weight compounds.

WADC TR 59-469, Part II

July 1961

SUBJECT: INVESTIGATION OF ORGANIC SEMICONDUCTORS
INVESTIGATOR: S. Aftergut, G. P. Brown

WADC TR 53-373 Sup 9

72

WADC TR 59-469, Part II (Continued)

CONTRACT: AF33(616)-6908, General Electric Co.
ABSTRACT: Measurements of the specific resistivity vs. temperature were performed on a variety of organic compounds including phenazine and some of its derivatives, solutions of phenazine in naphthalene, hydrogen-bonded compounds such as imidazole, benzimidazole, and 4-hydroxypyridine, pigments of the Aniline Black type, polymers, and pyrolyzed polymers. Activation energies for semiconduction were computed. The mechanism of semiconduction of organic compounds is discussed in terms of the overlap of molecular orbitals. The recent literature is reviewed.

WADD TR 60-178

April 1961

SUBJECT: RESEARCH ON HIGH-TEMPERATURE METALLO-ORGANIC COMPOUNDS AND POLYMERS
INVESTIGATOR: D. C. Bradley, I. M. Thomas
CONTRACT: AF61(052)-174, Birbeck College, University of London
ABSTRACT: New covalent dialkylamido-metal compounds of the type $M(NR_2)_x$ where M is Ti, Zr, V, Nb, or Ta; have been prepared from the metal chloride and a lithium dialkylamide. With Dialkylamido-compounds of Ti and Zr some or all of the dialkylamido-groups of one compound were replaced by another dialkylamido-group by reaction with a free secondary amine. Reactions of secondary dialkylamido-derivatives with primary amines gave rise to polymeric compounds containing M-N-M linkages. Ebullioscopic molecular weight measurements in benzene indicated the secondary dialkylamido-derivatives to be mainly monomeric; some were polymeric, due probably to co-ordination between the nitrogen atom of one molecule with the metal atom of a neighboring one. All compounds were violently hydrolysed by water, and gave alkoxides on reaction with alcohols.

WADD TN 60-271

June 1961

SUBJECT: USE OF DILATOMETRY FOR MEASURING POLYMERIZATION RATES
INVESTIGATOR: W. E. Gibbs, J. T. Murray
ABSTRACT: Techniques are presented and evaluated for the use of dilatometry in following polymerization reactions. Emphasis is placed on solution reactions where high precision is required with only small amounts (less than 25 milligrams) of monomer. Results are given for styrene, dimethyl-2,2'-dimethylene pimelate and methacrylic anhydride.

WADD TR 60-282, Part II

July 1961

SUBJECT: DETERMINATION OF THE RELATION BETWEEN
STRUCTURE AND RADIATION STABILITY OF
ARYL ETHER FLUIDS

INVESTIGATOR: G. E. Bohner, J. J. Schmidt-Collerus,
J. H. Weber

CONTRACT: AF33(616)-7220, Denver Research Institute

ABSTRACT: The polyphenyl ethers are of interest as
potential lubricants because of their demonstrated resistance
to thermal and radiolytic degradation.

Gamma radiolytic degradation of the aryl ethers forms polymers which are responsible for the observed viscosity increases. Higher molecular weight aryl ethers are more stable toward radiolysis than diphenyl ether. Diphenyl ether is degraded by polymerization reactions and by the reactions of the phenyl and phenoxy-free radicals resulting from carbon-oxygen bond scission. Radiolytically formed phenol results from the reaction of the phenyl radical at the ortho position of the ether. The phenoxy radical forms diphenoxybenzenes.

WADD TN 60-290

November 1961

SUBJECT: ORGANOMETALLIC COMPOUNDS OF GROUPS III,
IV, AND V OF THE PERIODIC TABLE, PROGRESS
REPORT I

INVESTIGATOR: C. Tamborski, G. Moore, F. Ford

ABSTRACT: The general reaction of an organometallic
or organometalloidal chloride with lithium in THF to yield
an organolithium compound is being studied. In this manner
organolithium compounds of C, Si, Ge, Sn, Pb, and P have
been prepared. The general formula of these organolithium
compounds is Ph_nMLi . An initial effort has been made to
investigate the direct procedure of making the alkylorgano-
lithium intermediates by the direct method. Thus far only
trimethyltin chloride has been studied. It does form a tri-
ethyltinlithium compound and is derivatized in very good
yields.

Various reactions of Ph_2CHLi and $\text{Ph}_3\text{-SnLi}$ have been carried out successfully. The preparation and reactions of the other M compounds of interest will be reported in progress reports to follow.

April 1961

SUBJECT: SYNTHESIS OF NITROGEN-CONTAINING AND HETEROCYCLIC FLUID SYSTEMS. Part II. The Preparation of Triazine, Quinoline and Tertiary Amino Derivatives

INVESTIGATOR: E. H. Kober, B. F. Clark, W. J. Schnabel, H. A. Schroeder

CONTRACT: AF33(616)-6342, Olin Mathieson Chemical Corp.

ABSTRACT: This report presents the results of an investigation toward the synthesis of nitrogen-containing and heterocyclic compounds as potential thermally stable base stock fluids. Work on substituted derivatives of melam and melem, earlier carried out under this contract and reported in WADD TR 60-315, was continued. It is concluded that in these two classes of compounds thermal stability approaching the 1000°F goal can only be obtained with substituents which impart very high melting points to the resulting compounds. Further investigations were concerned with the synthesis of substituted s-triazines, quinolines, benzo(h)-quinolines, and silicon-containing aromatic tertiary amines. Many preparations showed melting points below 100°C, some had good thermal stability at 800°F, but only few exhibited fair stability at 900°F which appears to be the upper thermal stability limit of these compounds.

April 1961

SUBJECT: THE PREPARATION OF MONO-AND DI-SUBSTITUTION PRODUCTS OF TRICHLORO-symm-TRIAZINE BY REPLACEMENT OF CHLORINE BY TRIALKYLSILYLPHENYL

INVESTIGATOR: H. W. Post, C. J. Murphy

CONTRACT: AF33(616)-6480, University of Buffalo

ABSTRACT: The following have been prepared and characterized: 2,4-dichloro-6-(p-trimethylphenyl)-s-triazine, 2,4-dichloro-6-(p-tolyl)-s-triazine and 2-chloro-4,6-diphenyl-s-triazine. In addition, as intermediates, allyldimethyl-p-bromophenylsilane, benzyldimethyl-p-bromophenylsilane, vinyl-dimethyl-p-bromophenylsilane, amyldimethyl-p-bromophenylsilane, and trimethyl-p-bromophenylsilane have been prepared and characterized with the intention of using these in the preparation of other triazine derivatives.

December 1960

SUBJECT: PHOTODEGRADATION OF HIGH POLYMERS, PART
I. The Homogeneous Ionic Polymerization
of α -Methylstyrene

INVESTIGATOR: R. B. Fox, L. Isaacs

CONTRACT: MIPR(33-616)-61-10, Naval Research Lab

ABSTRACT: A practical method has been developed
for the laboratory preparation of poly- α -methylstyrene.
The procedure involves the reaction of α -methylstyrene with
sodium in tetrahydrofuran solution at a temperature above the
ceiling temperature for the monomer concentration used and
propagation by cooling to -78°C . All operations are con-
ducted under helium. Optical densities of the solutions
just prior to cooling are related empirically to the intrinsic
viscosities of the polymers produced, and viscosities are
given as a function of polymerization time for a fixed so-
lution optical density. Relatively monodisperse polymers
with predictable molecular weights in the range of 50,000
to 1 million were prepared. Their molecular weight dis-
tributions were compared qualitatively by sedimentation
methods with those obtained by heterogeneous sodium initiation
and boron trifluoride etherate initiation.

December 1961

SUBJECT: PHOTODEGRADATION OF HIGH POLYMERS, PART
II. Vacuum Photolysis of Poly- α -
Methylstyrene

INVESTIGATOR: S. Stokes, R. B. Fox

CONTRACT: MIPR (33-616)-61-10, Naval Research Lab

ABSTRACT: The photodegradation of thin films of
poly- α -methylstyrene by near ultraviolet light has been
studied in vacuum at 27° and 115°C . Changes taking place
during photolysis were followed by determining monomer forma-
tion by ultraviolet spectroscopy, residue molecular weight
through a measurement of intrinsic viscosity, and volatile
products by mass spectroscopy. Quantum yields for chain
scission and monomer formation were calculated. The main
features of the photolysis were found to be random chain
scission in the polymer with subsequent depolymerization of
the radicals formed to yield only small amounts of monomer;
the kinetic chain length is on the order of 15 in the tempera-
ture range studied.

WADD TR 60-865

March 1961

SUBJECT: TETRAALKYLSILANES: WIDE LIQUID RANGE
FLUIDS

INVESTIGATOR: G. Baum, C. Tamborski

ABSTRACT: Tetraalkylsilanes were investigated as potential lubricating fluids. Methods of synthesis, purification and analysis are discussed. Dodecyltrialkylsilanes, didodecyldialkylsilanes and cyclopentamethylenedialkylsilanes were prepared. The alkyl groups ranged from methyl to octadecyl. Effect of molecular structure on pour point and viscosity is discussed. The fluids are stable up to about 600°F. Wear characteristics of the unblended fluids were investigated in the four-ball wear machine. Oxidation resistance of the fluids are moderate at 500°F.

WADD TR 60-911

April 1961

SUBJECT: RESEARCH ON INORGANIC POLYMER SYSTEMS

INVESTIGATOR: A. L. McCloskey, W. G. Woods, R. J.
Brotherton

CONTRACT: AF33(616)-7303, U.S. Borax Research Corp.

ABSTRACT: This report includes investigations on the chemistry of thermally stable inorganic and semi-inorganic polymer systems showing promise of stability at high temperatures. Systems based on B-B, B-N, Al-O and Al-N bonding have been studied as well as polymers containing tin, various pi-bonded systems and alternate approaches to inorganic polymer systems.

WADD TR 60-912

May 1961

SUBJECT: PREPARATION AND PROPERTIES OF SOME
POLYETHERS

INVESTIGATOR: G. F. L. Ehlers

ABSTRACT: A number of polyethers of the type
 $(-\text{CH}-\text{CH}_2-\text{O}-)_n$

R have been prepared by ionic catalysis of the corresponding epoxy-compounds and identified by elementary analysis, infrared spectra, inherent viscosity and molecular weight. The relative thermal stability of these polymers has been determined and indicated the superiority of phenyl substituted polyethers before those with fluorinated side chains.

ASD TR 61-1, Part I

July 1961

SUBJECT: THE PREPARATION OF ORGANOMETALLIC
DERIVATIVES OF INORGANIC "BENZENOID"
COMPOUNDS

INVESTIGATOR: D. Seyferth, W. Freyer, G. Raab

CONTRACT: AF33(616)-7124, Mass. Inst. of Tech.

ABSTRACT: The attempt to prepare silyl-substituted
phosphazenes by the reaction of silyl-substituted tetra-
chlorophosphoranes with ammonium chloride was not successful
because one of the key reactions - that between trimethyl-
silylmethyldichlorophosphine and chlorine failed to go in
the desired direction; chlorinolysis of the silicon-carbon
bond, rather than formation of trimethylsilylmethyltetra-
chlorophosphorane, was observed. A successful synthesis
of the hitherto unknown reagent, perfluoroxinyl lithium, has
been developed. The exchange reaction occurring between
phenyl- and n-butyllithium and perfluorovinyltin compounds
in ether or in pentane gives perfluorovinyl-lithium in 40-
45% yield. B-chloro-B', B''-bis(trimethylsilylmethyl)-
N,N',N''-trimethylborazene was prepared in 47% yield. This
compound will serve as an intermediate in future studies.

ASD TR 61-1, Part II

March 1962

SUBJECT: THE PREPARATION OF ORGANO-METALLIC
DERIVATIVES OF INORGANIC "BENZENOID"
COMPOUNDS

INVESTIGATOR: D. Seyferth, M. Takamizawa, H. Yamazaki

CONTRACT: AF33(616)-7124, Mass. Inst. of Tech.

ABSTRACT: Selected addition reactions of B-trivinyl-
N-triphenylborazine were investigated. Addition of bromo-
trichloromethane, carbon tetrabromide, benzenethiol, hydrogen
bromide, dimethylchlorosilane and methyldichlorosilane to
this borazine is described. Phenyllithium cleaved this vinyl-
borazine, giving triphenylborane and aniline. The first
carbon-bridged bisborazine, 1,4-bis (B-bis(trimethylsilyl-
methyl)-N-trimethyl-(B)-borazyl)butane, has been prepared
and its thermal and hydrolytic stability studied. The com-
plete hydroboration of trimethylvinylsilane and allyltri-
methylsilane, as well as partial hydroboration of the former,
are described. Use of the boranes produced in borazine
syntheses has been studied.

ASD TR 61-2

June 1961

SUBJECT: SYNTHESIS OF INORGANIC POLYMER HIGH
TEMPERATURE FLUIDS
INVESTIGATOR: G. M. Nichols
CONTRACT: AF33(616)-7158, Eastern Laboratory, E. I.
DuPont

ABSTRACT: Reactions of PCl_5 and NH_4Cl with a variety of metal halides have yielded materials composed of a (PNCl_2) in portion linked to the metal halide. Most of these compositions are liquids with a wide liquid range and pyrolytic stability at 1100°F . Metal halides found to exert this effort are SbCl_5 , SbCl_3 , AlCl_3 , AlBr_3 , BCl_3 , BF_3 , TiCl_4 , and ZnCl_2 . All of the modified products are sensitive to hydrolysis and their exact structures have not been rigorously established. Viscosity measurements from 0 to 700°F indicate the metal halide stabilized phosphonitrilic chloride liquids have satisfactory temperature-viscosity characteristics.

Aryloxy derivatives of cyclic phosphonitrilic chloride polymers have been prepared. These compounds, which vary from crystalline solids to viscous liquids, have good hydrolytic stability, very low vapor pressures, and thermal stability to approximately 750°F .

ASD TR 61-4

August 1960

SUBJECT: RESEARCH AND DEVELOPMENT OF HIGH TEMPERA-
TURE STABLE ORGANO-PHOSPHORUS COMPOUNDS
INVESTIGATOR: C. F. Baranauckas, R. D. Carlson, E. E.
Harris, R. J. Lisanke
CONTRACT: AF33(616)-7191, Hooker Chemical Corp.
ABSTRACT: A series of alkylene and arylenebisdi-
phenyl-phosphines and the corresponding
bisphosphine oxides have been prepared by modification of
existing synthetic methods.

Thermal testing by a weight loss and chemical change technique has been carried out at $300\text{--}450^\circ\text{C}$. in a nitrogen atmosphere. The arylenebisphosphines and bisphosphine oxides are more stable than alkylene, with the tri- and tetra-methylene and the neopentylene being the most stable alkylene bridges. The aromatic series appears to begin change by losing ring hydrogen with subsequent ring condensation. The alkyls all seem to produce P-O-R structures or, in the case of phosphines, P-H and products derived therefrom.

ASD TR 61-4 (Continued)

A thermal study of simple arylphosphines and arylphosphine oxides and sulfides has yielded some clues to decomposition routes, which with the above data allow some tentative suggestions on mechanisms to be made.

WADD TR 61-12

April 1961

SUBJECT: HIGH POLYMERIC MATERIALS
INVESTIGATOR: C. S. Marvel, T. Moeller, J. C. Bailar, Jr.
CONTRACT: AF33(616)-5486, University of Illinois
ABSTRACT: A series of polyaromatics has been studied. No polyquinoline has been obtained. All of the other types show excellent thermal stability. The polymer prepared from diphenyl isophthalate and 3,3'-diaminobenzidine has excellent thermal and hydrolytic stability and attractive physical properties such as tensile, modulus, and elongation when tested in thin films. Aminolysis of tetrameric phosphonitrilic chloride yields di-, tetra-, or octasubstituted products, depending upon conditions. Synthesis of phosphonitrilic bromides have been improved. The trimeric and tetrameric fluorides have been obtained by methathesis in nitrobenzene. Metallated organic polymers have been prepared from bis-salicylaldehydes and metallated polyamines. Complexes of salicylidene-ethanolamine were found to be unsuitable for preparation of thermally stable polymers. Coordination polymers of bis-(β -diketones) are shown to exhibit a variety of bond types. Polymers made by condensation of polyhydroxy compounds with silicon and aluminum chlorides are thermally unstable.

ASD TR 61-16

June 1961

SUBJECT: MOLECULAR STRUCTURE AND PHYSICAL BEHAVIOR OF POLYMERS
INVESTIGATOR: M. Morton, F. Bueche, S. D. Gadkary, T. E. Helminiak
CONTRACT: AF33(616)-6965, University of Akron
ABSTRACT: Linear polystyrenes with reactive end groups and having M_w/M_n 1.10 have been prepared. These molecules have been linked into star-type molecules by use of silicon tetrachloride as linking agent. The resultant branched polymer is found to consist of both tri- and tetra-branched polymer stars. A small amount of polymer having twice the original molecular weight may also be

ASD TR 61-16 (Continued)

present. Some linear primary polymer also remains. These species have been fractionated into well defined groups so that tetra-branched and tri-branched stars were obtained. Preliminary measurements of the intrinsic viscosity in toluene indicate that the ratio of the intrinsic viscosity of tetra-star molecules to that of a linear polymer of the same molecular weight is about 0.89, whereas for tri-star molecules it is 0.94. There is some indication that the anionic polymers prepared in benzene do not obey the same intrinsic viscosity - molecular weight equation as that commonly used for free radical polymers. Calculations are given which show how the molecular weight distribution of the linear molecules influences the molecular weight distribution of the branched polymer. The weight-average molecular weight of a new polymer, PB-1, poly-m-phenylene-dibenzimidazole, prepared by Professor C. S. Marvel was determined, and an estimate of its size in solution was made. It was found to be an extremely stiff, extended chain. Creep measurements have been made on carbon black filled SBR, styrene-butadiene rubber, at very high stresses. It is found that to a first approximation the creep and recovery are related by the Boltzmann force superposition theory although discrepancies do appear. The permanent set observed is small even at very high elongations.

ASD TR 61-22

SUBJECT: POLYMER STRUCTURES AND PROPERTIES
INVESTIGATOR: E. F. Casassa, T. A. Orofino, J. W. Mickey, T. G. Fox, G. C. Berry, D. J. Plazek, R. E. Kerwin, H. Nakayasu, V. R. Allen, C. A. J. Hoeve, M. O'Brien
CONTRACT: AF33(616)-6968, Mellon Institute
ABSTRACT: Investigations relating to dilute solutions of polymers have comprised: a theoretical treatment of Rayleigh scattering to include both intramolecular and intermolecular optical interference effects; the temperature dependence of the second virial coefficient for polystyrene in cyclohexane; the intrinsic viscosity-molecular weight relation for poly-(vinyl acetate) in butanone; and design and construction of a precision light scattering photometer. The melt viscosity-molecular weight relation at 218° for monodisperse polystyrene prepared anionically was found to be identical with that for ordinary fractions.

Poly-(vinyl acetate), prepared by an

ASD TR 61-22 (Continued)

emulsion polymerization in the presence of a protein, yielded an insoluble component containing bound protein.

Preliminary stress-strain measurements on cross-linked swollen polyethylene gave only equivocal evidence for specific solvent effects on the unperturbed random-flight dimensions of the polymer chains.

WADD TR 61-52

May 1961

SUBJECT: RESEARCH ON HIGH-TEMPERATURE METALLO-ORGANIC COMPOUNDS AND POLYMERS
INVESTIGATOR: D. C. Bradley, I. M. Thomas, E. G. Torrible
CONTRACT: AF33(616)-6934, University of Western Ontario
ABSTRACT: Dialkylamido-derivatives of vanadium, niobium, tantalum, chromium, molybdenum and tungsten have been synthesized by reactions involving lithium dialkylamides and metal chlorides. Only the dimethylamido-, N-methyl n-butylamido-, and piperidino-groups gave stable pentakis-derivatives of Nb and Ta. With R_2N groups ($R = Et, Pr^i$ or Bu^i) the pentakis-derivatives decomposed to stable $Nb(NR_2)_4$ and $RN-Ta(NR_2)_3$ compounds. With molybdenum and tungsten, $Mo(NEt_2)_4$ and $W(NEt_2)_4$ were isolated. Some remarkable new compounds of Mn, Fe, Co and Ni were isolated from the $LiNEt_2$ -metal chloride reactions. Preliminary work involving reactions of $Ti(NMe_2)_4$ or $Ti(NEt_2)_4$ with primary amines resulted in the formation of polymeric compounds of which those derived aniline and p-phenylene diamine were the most stable.

WADD TR 61-57

April 1961

SUBJECT: SYNTHESIS OF THERMALLY STABLE DIELECTRIC RESINS CONTAINING HETEROCYCLIC ORGANIC RING SYSTEM
INVESTIGATOR: P. E. Brumfield, A. Lebovits, B. Rudner, S. C. Temin
CONTRACT: AF33(616)-7021, Koppers Company, Inc.
ABSTRACT: This report covers synthetic studies designed to lead to polymeric organic compounds of high thermal stability with application in forming high temperature dielectric materials. Two approaches are being followed; namely, the formation of polymers containing aromatic nuclei joined through heterocyclic groups and the polymerization of

WADC TR 61-57 (Continued)

fluoroolefins from monomers having a pendant heterocyclic substituent. The initial phase of study of polymer chains incorporating heterocyclic groups consisted of preparing model thiazole derivatives for thermal stability testing. On the basis of incomplete results, heat stability tests indicate that the benzothiazole structure and the linkage through the 2,4-positions of the thiazole ring give the more heat stable structures. A study of the applicability of a Primuline type of reaction to polymer formation is in progress. The fluoroolefin study was aimed at the synthesis of monomers of the type $R-CF=CF_2$, where R was a stable heterocyclic ring; such as, pyridyl, thiazolyl, imidazolyl or carbazolyl. Synthetic approaches consisted of the reaction between organolithium compounds and tetrafluoroethylene, the reaction between organolithium compounds and tetrafluorodibromoethane, and the attempted dehydrofluorination of N-(1,1,2,2-tetrafluoroethyl) carbazole. None of these were successful.

WADC TR 61-79, Part I

April 1961

SUBJECT: RESEARCH ON PHOSPHORUS-CONTAINING AND OTHER INORGANIC AND SEMI-ORGANIC POLYMERS
INVESTIGATOR: E. C. Chapin, J. W. Dale, I. B. Johns, M. L. Nielsen, J. R. Van Wazer, R. J. Wineman
CONTRACT: AF33(616)-6950, Monsanto Chemical Co.
ABSTRACT: A program involving eight separate approaches to the preparation of useful inorganic and semi-organic polymers with extreme thermal stability for each area of research as follows: I. Phosphorus Polymers; II. Cyanofluorocarbons; III. Conjugated Fluorocarbon Polymers; IV. Superpressure Studies; V. Organophosphorus Polymers; VI. Copolymers from Nitrilic Chlorides and Oxychlorides; VII. Polymers from Dichlorophosphorus Compounds and Diamines; and VIII. Synthetic Macromolecules Containing Ionic Binding. Conclusions and recommendations for further work on each phase are given.

WADD TR 61-79, Part II

February 1962

SUBJECT: RESEARCH ON PHOSPHORUS-CONTAINING AND OTHER INORGANIC AND SEMI-ORGANIC POLYMERS
INVESTIGATOR: E. C. Chapin
CONTRACT: AF33(616)-7853, Monsanto Research Labs
ABSTRACT: A program involving eight distinct

WADD TR 61-79, Part II (Continued)

approaches to preparation of thermally stable inorganic and semi-organic polymers was continued. Results are reported separately for each approach as follows: I. Phosphorus Polymers; II. Cyanofluorocarbons; III. Conjugated Fluorocarbon Polymers; IV. Superpressure Studies; V. Organo-Phosphorus Polymers; VI. Polymers from Difunctional Phosphorus Compounds and Amines; VII. Copolymers from Nitrilic Chlorides and Oxychlorides. The Ammonolysis of Mixed Covalent Metal Halides; and VII. Polymers Containing Phosphorus-Carbon Bonds.

WADD TR 61-84

May 1961

SUBJECT: EMPIRICAL METHODS FOR CALCULATION OF BOND ENERGIES
INVESTIGATOR: H. H. Jaffe, Van Tran Zung
CONTRACT: AF33(616)-6900, University of Cincinnati
ABSTRACT: Slater parameters (F's and G's) were calculated for the elements scandium to bromine and are tabulated. These parameters were used to calculate the energies of a wide variety of valence states of these elements, and of their unipositive ions, and hence valence state ionization potentials are derived. These data are tabulated.

WADD TR 61-139

May 1961

SUBJECT: THE EFFECT OF CONCURRENT STRAINING AND A 1-PERCENT MAGNESIUM ADDITION ON THE RECOVERY BEHAVIOR OF ALUMINUM
INVESTIGATOR: T. E. Tietz, C. L. Meyers, J. L. Lytton
CONTRACT: AF33(616)-7156, Lockheed Aircraft Corp.
ABSTRACT: The effect of elastic strain, concurrent creep strain, and a 1-percent magnesium addition on the recovery behavior of a high-purity aluminum was investigated. The degree of recovery of the prestrained test material was measured in terms of tensile flow stress at room temperature after recovery treatments between 80° and 200°C. Recovery behavior under no-load conditions was evaluated for the 99.995-percent aluminum and the Al-1%Mg alloy. The effect of concurrent elastic strain and creep straining during recovery of 99.995 percent aluminum was also studied. The activation energy for recovery of the 99.995-percent aluminum between 80° and 200°C was found to be $23,300 \pm 2,000$ calories per mole, and the activation energy for the

WADD TR 61-139 (Continued)

Al-1%Mg alloy was 27,500 calories per mole. Whereas concurrent elastic strain was concluded to have no effect on the rate of recovery of the 99.995-percent aluminum in terms of flow stress, concurrent creep straining had a very significant effect. However, the activation energy of the recovery process was not found to be significantly different as a result of concurrent creep straining.

ASD TR 61-172

February 1962

SUBJECT: RESEARCH ON ORGANIC CHEMISTRY AND FERROCENES
INVESTIGATOR: K. Schlogl
CONTRACT: AF61(052)-383, University of Vienna
ABSTRACT: Various bridged ferrocenes, bistrimethylene and diethyltrimethylene ferrocene and acetylated derivatives therefrom were prepared and their stereochemistry elucidated. Several ferrocenyl ethynylcarbinols were synthesized from the appropriate aldehyde or acetyl derivative. Oxidative coupling of ferrocenylethynylcarbinols gave the corresponding diacetylenic glycols. Hydrogenation of the secondary alcohols led to saturated ferrocenyl carbinols and glycols while tertiary compounds by cleavage of the C-O-bond underwent simultaneous hydrogenolysis to give ferrocenyl alkanes. Diferrocenylcarbinol gave diferrocenylmethane. Saturated ferrocenyl carbinols and glycols were easily dehydrated with acid alumina to yield ferrocenyl alkenes while compounds of suitable structure gave ethers. Diferrocenyldimethyl ether was obtained from hydroxymethylferrocene, while heteroannular glycols gave bridged ethers by cyclo dehydration.

ASD TR 61-237, Part I

August 1961

SUBJECT: THE PREPARATION OF CERTAIN HETEROCYCLIC POLYMERS BY AN ALTERNATING INTRAMOLECULAR-INTERMOLECULAR CHAIN PROPAGATION
INVESTIGATOR: G. B. Butler, C. F. Hauser, C. Rushing, W. C. Bond, Jr.
CONTRACT: AF33(616)-6887, University of Florida
ABSTRACT: A number of additional monomers containing phosphorus and silicon have been synthesized and their polymerization studied. These monomers contain two non-conjugated double bonds, either in the 1,4-, 1,5-, 1,6-, or 1,7-positions. Copolymerization of the 1,4-dienes with monoolefinic monomers has been studied. The 1,5-, 1,6-,

ASD TR 61-237, Part I

1,7-dienes are capable of forming five- six- or seven-membered rings, respectively, and relative tendencies of certain of these monomers to close the rings have been prepared. Poly(diallyldiphenylsilane) has been fractionated, molecular weights determined on the various fractions by light scattering measurements and the molecular weight distribution determined. Preliminary studies on copolymerization of tetravinyl silane and tetravinyl tin with certain monoolefinic monomers indicates that these monomers are capable for forming linear, soluble, saturated polymers containing bicyclic or tricyclic units in the chain.

WADD TR 61-255

June 1961

SUBJECT: SYNTHESIS AND EVALUATION OF THERMALLY STABLE POLYMERS
INVESTIGATOR: G. P. Brown, A. Goldman, C. D. Doyle
CONTRACT: AF33(616)-7076, General Electric Co.
ABSTRACT: Poly-m-phenoxyene has been synthesized by self-condensation of alkali metal salts of m-bromophenol condensation of salts of resorcinol with m-dibromobenzene and self-condensation of resorcinol. A single sample of poly-m-phenoxyene was tested via thermogravimetric analysis and was shown to possess outstanding thermal stability. Polymer evaluation was made using kinetic analysis of thermogravimetric data, illustrated for zero-order(octamethylcyclotetrasiloxane) and first-order (polytetra-fluoroethylene) volatilization; effects of varying sample geometry in thermogravimetry; and thermo-particulate analysis as a means of observing incipient and vestigial volatilization, heat-softening in powdered samples, as well as such underlying causes of heat-softening as relaxations and thermodynamic transitions, differential thermal analysis, volume dilatometry, x-ray and electron diffractometry, methods of observing mechanical, electrical and nuclear magnetic relaxation effects, and a new heat-softening test. As a part of the study of nuclear magnetic resonance, some styrene-divinylbenzene copolymers of various degrees of cross-linking are compared.

ASD TR 61-409

October 1961

SUBJECT: MECHANISM OF ALTERNATING INTER-INTRA MOLECULAR PROPAGATION I. METHACRYLIC ANHYDRIDE

WADC TR 53-373 Sup 9

86

ASD TR 61-409 (Continued)

INVESTIGATOR: W. E. Gibbs, J. T. Murray
ABSTRACT: The solution polymerization of methacrylic anhydride, a monomer which undergoes predominantly inter-intramolecular propagation, shows kinetics similar to vinyl polymerization. The over-all rate of polymerization is proportional to the square-root of the initiator concentration and to the $3/2$ power of the monomer concentration. The apparent, overall energy of activation was found to be 23.0 kcal/mole, when azobis-isobutyronitrile was used as the initiator. The value of $E_p - E_t/2$ obtained is 8.0 kcal/mole. This compares with the value of $E_p - E_t/2$ found for methacrylic acid in the same solvent (DMF) of 9.8 kcal/mole. Both values are in the proximity of values for other vinyl monomers.

ASD TR 61-443

September 1961

SUBJECT: POLYMERIZATION OF N,N-DIALLYLMELAMINE
INVESTIGATOR: W. E. Gibbs, R. L. VanDeusen
ABSTRACT: The polymerization of N,N-diallylmelamine in the solid state has been found to yield soluble, essentially linear, relatively low molecular weight polymer. The polymerization in the solid state is suggested to proceed by an alternating intra-intermolecular propagation reaction previously known to occur only in solution reactions. Preliminary kinetic results indicate an overall activation energy for the post-irradiation reaction of 15.5 ± 1.5 kcal/mole. This is in the proximity of values already reported for the solid state polymerization of acrylamide. Polymerization of diallylmelamine was also effected in solution using free radical initiation. The polymer obtained was nearly identical to that obtained in the solid state reactions. The \bar{M}_w of one polymer was found to be $7,500 \pm 1,000$ grams/mole.

ASD TR 61-561

March 1962

SUBJECT: KINETIC STUDY OF IONSENSITIZED TERMINATIONS OF RADICAL POLYMERIZATION
INVESTIGATOR: E. A. S. Cavell
CONTRACT: AF61(052)-376, University of Southampton
ABSTRACT: For polymerizations of acrylamide at 25.0°C , initiated by 4-4'-azo-bis-4-cyanopentanoic acid, $R_p \propto (m_1)^{1/2}$ up to 10% conversion. In the presence of ferric perchlorate and decimolar perchloric acid, the rate of polymerization is reduced and $R_p \propto (\text{Catalyst})/(\text{Fe}^{3+})$.

ASD TR 61-622

February 1962

SUBJECT: THERMOGRAVIMETRIC ANALYSIS OF POLYMERS
INVESTIGATOR: G. F. L. Ehlers
ABSTRACT: Tests of 176 conventional and experimental polymers were made in a Chevenard thermo-balance under nitrogen up to 900°C; a number of these also were tested under air. The results were correlated to the structure of the polymers. The effect of crosslinking, symmetry, branching, presence of active hydrogen, etc., on the thermal stability are given, and breakdown temperatures for aliphatic, perfluorinated and aromatic systems are presented together with those of various types of linkage.

Isothermal weight loss studies of several polymers under nitrogen and under air were correlated to the TGA curves, obtained at constant heating rate.

ASD TDR 62-72

January 1962

SUBJECT: LITERATURE SURVEY AND BIBLIOGRAPHY;
ULTRAHIGH-PRESSURE EFFECTS ON ORGANIC,
INORGANIC, AND SEMI-ORGANIC MATERIALS
INVESTIGATOR: R. A. Mayer, R. I. Leininger
CONTRACT: AF33(616)-7471, Battelle Memorial Inst.
ABSTRACT: A literature survey from 1926 to December 1961 on pressure effects greater than 20,000 atms. on inorganic, organic and semi-organic materials is given.

It was found that research has been directed primarily toward the physical transitions at the high pressures with less emphasis on the chemical changes. However, in a number of instances, chemical reactions did occur because of high pressures. Examples for inorganics are; decomposition of metal salts and oxides, reduction of oxides to free metals, and irreversible changes in glasses.

In organics the most significant findings are that some normally unreactive compounds such as tetra-substituted ethylenes can be polymerized at high pressures and that some polymers gelled under these conditions. Aromatic nitriles can be trimerized to triazines. Other compounds such as cellulose, Neoprene, benzamide, and benzaldoxime were found to be irreversibly changed by the drastic conditions.

ASD TDR 62-73

January 1962

SUBJECT: EFFECTS OF ULTRAHIGH PRESSURES ON THE
FORMATION AND PROPERTIES OF ORGANIC,
SEMI-ORGANIC AND INORGANIC MATERIALS
INVESTIGATOR: E. J. Bradbury, H. H. Krause, C. B.
Sclar
CONTRACT: AF33(616)-7471, Battelle Memorial Inst.
ABSTRACT: The effect of ultrahigh pressures (up
to 90,000 atm) was studied on organic polymers and a variety
of semiorganic and inorganic materials.

Results on organic polymers indicate that the effect of compression is influenced by the compression attained, the compression rate, the holding period, and the polymer used. The most influential parameters within the target range of compression appear to be the polymer and the holding period. No generalized pattern of behavior for polymers was noted.

Ultrahigh-pressure high-temperature studies of heteropolynuclear acids (2) and salts (2) appear to have pressure dependent modifications over a broad pressure-temperature range of 75,000 atm and 1300°C.

ASD TDR 62-200, OTS Release

January 1962

SUBJECT: SYNTHESIS OF THERMALLY-STABLE POLYMERS
INVESTIGATOR: C. S. Marvel, J. J. Bloomfield, D. A.
Frey
CONTRACT: AF33(616)-7908, University of Arizona
ABSTRACT: A synthesis has been devised which produces pure diacrylylmethane in good yields.

Three new soluble polybenzborimidazolines have been prepared by the condensation of 3,3'-diaminobenzidine with the tetrabutyl esters of phenyl-1,3-diboronic acid, phenyl-1,4-diboronic acid and ferrocene-1,1'-diboronic acid, respectively. Thermogravimetric analysis curves indicate that the phenyl boronic acids polymers are stable to over 500°C whereas the ferrocene derivative decomposes at about 350°C.

Further work has been done on the polymerization of 1,3-cyclohexadiene. Improved dehydrogenation procedures have been devised for converting poly-1,3-cyclohexadiene linear polyphenyl.

ASD TDR 62-200 (Continued)

Poly-2,2'-(m-phenylene-5,5'-bibenzimidazole) has been converted to derivatives having cyanomethyl and cyanoethyl groups in place of the imine hydrogen atom.

ASD TDR 62-209, OTS Release

February 1962

SUBJECT: HIGH POLYMERIC MATERIALS
INVESTIGATOR: T. Moeller, J. C. Bailer, Jr.
CONTRACT: AF33(616)-7918, University of Illinois
ABSTRACT: Polymerization of the phosphonitrilic isothiocyanates occurs readily. The phosphonitrilic bromides react with both Grignard reagents and organolithium compounds, but the intermediates thus formed have not yet been composed to yield reasonable quantities of organophosphonitrilic derivatives. The syntheses of trimeric and tetrameric phosphonitrilic fluorides by metathetical reactions of the chlorides with sodium fluoride in nitrobenzene have been effected. A new reaction between the completely fluorinated compounds and aryllithium compounds gives stereoselective substitution of the fluorine atoms by aryl groups.

Section II - (1) Attempts to link β -diketone complexes together have failed because substituent groups on the chelate rings are unreactive. (2) Triethylenediamine has been shown to coordinate through both of its nitrogen atoms, and thus to form polynuclear complexes. (3) The compositions, solubilities, and heat stabilities of a number of complexes of bis- β -diketones with bivalent, 4-covalent metals have been studied.

ASD TDR 62-252

March 1962

SUBJECT: SYNTHESIS OF POLYHETEROCYCLICS
INVESTIGATOR: P. E. Brumfield
CONTRACT: AF33(616)-7021, Koppers Co., Inc.
ABSTRACT: Two approaches to the synthesis of organic polymers having high thermal stability were followed: condensation polymerization to form arylene heterocyclic chains and addition polymerization of perfluorovinyl compounds to form fluorocarbon chains with pendant heterocycles.

Six different types of polymeric compounds were prepared in which the polymer chain contained arylene and thiazole units. The products were dark infusible powders.

ASD TDR 62-252 (Continued)

Their thermal stabilities were determined as a guide for further synthesis.

Synthesizing perfluorocarbon polymers with pendant heterocyclic groups first requires the preparation of monomers with the structure $\text{RCF}=\text{CF}_2$, where R is a stable heterocyclic ring. Several methods designed to produce the monomers have been examined without success.

ASD TDR 62-322

March 1962

SUBJECT: PHENYLENE SULFIDE POLYMERS
INVESTIGATOR: H. A. Smith, C. E. Handlovits
CONTRACT: AF33(616)-7251, Dow Chemical Co.
ABSTRACT: The investigation of phenylene sulfide polymers has been divided into four areas of endeavor. These are a kinetic study of a model reaction, monomer synthesis, polymerization, and determination of polymer properties. From the results of the kinetic study and monomer synthesis coupled with the early results in the other two fields cuprous p-bromothio-phenoxide has been chosen as the monomer of choice for the preparation of linear phenylene sulfide polymers. Later work on this monomer has shown that a number average degree of polymerization greater than 400 can be obtained either by solid state or solution polymerization. This polymer which has useful polymeric properties is stable in air or nitrogen to 450°C and forms a polymeric residue stable to 900°C under nitrogen.

ELASTOMERS

WADD TR 55-492, Part VI

April 1961

SUBJECT: HIGH TEMPERATURE FUEL RESISTANT RUBBER COMPOUNDS
INVESTIGATOR: R. G. Spain, K. L. Paciorek, W. G. Lajiness, B. A. Merkl
CONTRACT: AF33(616)-5544, Wyandotte Chemicals Corp.
ABSTRACT: A study was made of vulcanization processes of fluorinated elastomers.

Several model compounds were prepared and their reactions with various amines were examined. Dehydrohalogenation constituted the first step; primary and secondary amines added to the unsaturated centers; mono and diamino compounds were isolated.

Differential thermal analysis showed the untreated fluoroelastomers to be more thermally stable than the vulcanized materials. Thermal decomposition of fluorinated compounds proceeded with large energy release.

Evaluations of experimental vulcanizates were conducted in fuels (JP-6 and HTF) and air at elevated temperatures (400°-750°F). Amine and peroxide curing agents were assessed.

WADC TR 56-272, Part VI

May 1961

SUBJECT: DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS
INVESTIGATOR: W. R. Hickman, R. J. Williams, G. E. Trepus, R. S. Roper, J. R. Conroy, R. G. Elliot
CONTRACT: AF33(616)-5722, Boeing Airplane Co.
ABSTRACT: The purpose of this present study was to obtain and develop seal design information so that a handbook could be prepared concerning seals for extreme and/or unusual environments. A survey of literature concerning seals and seal materials was conducted with an emphasis placed on seals for extreme environments and on the aging of seals and its effects. Physical properties, including thermal expansion, compression modulus, tensile strength, elongation, and modulus, tensile and compression stress relaxation, compression set, and frictional characteristics, were determined at temperature extremes in the range of -425°F

WADC TR 53-373 Sup 9

92

WADC TR 56-272, Part VI (Continued)

to 600°F. Functional tests reported include static and dynamic seal tests at temperatures from room temperature to 1000°F, hydraulic rod seal tests at temperatures from -65°F to 800°F at pressures from 1000 to 4000 psig, and static and dynamic seal tests at cryogenic temperatures. Aging tests, both accelerated and natural, were conducted.

The property found to be most important in determining the effects of aging on elastomeric seal materials was compression set. Certain properties were found to be important in the choice of seal materials for specific conditions.

WADC TR 59-428, Part II

April 1961

SUBJECT: DESIGN HANDBOOK FOR O-RINGS AND SIMILAR ELASTIC SEALS
INVESTIGATOR: W. R. Walker
CONTRACT: AF33(616)-5722, Boeing Airplane Co.
ABSTRACT: This is a continuation of the summation of work accomplished under Air Force Contract AF33(616)-5722, Task No. 73810. It is presented as Part II of a handbook covering the mechanism of O-ring sealing. Data presented herein is concerned with hydraulic and pneumatic systems utilizing static and dynamic type seals at temperatures exceeding 275°F and for static applications at cryogenic temperatures. The effect of vibration, pressure cycling, seal materials and fluids on the operational efficiency of seals and back-up rings are discussed. Sections are devoted to cryogenic, fuel, and, vacuum system seals, and aging and age control. It also includes physical properties of elastomers at high temperatures and a bibliography.

WADD TN 60-300

April 1961

SUBJECT: COMPATIBILITY OF ELASTOMERS WITH FUEL ANTI-ICING ADDITIVE
INVESTIGATOR: P. A. House
ABSTRACT: Fuel anti-icing additives have proved helpful in eliminating ice in aircraft fuel systems. One such additive was flight tested until it was found that a coating material used in integral fuel tanks was deteriorating. Available laboratory tests did not predict this development. A study of the non-correlation between laboratory and flight

WADD TN 60-300 (Continued)

test data is given in this report, as well as an investigation of the compatibility of all aircraft elastomeric materials with this additive. It was found that the particular additive in its present form cannot be used due to the lack of compatibility but evaluation methods are given for future compatibility evaluation of anti-icing additives.

WADD TR 60-922, Part I

March 1961

SUBJECT: STRESS RELAXATION OF ELASTOMERS
INVESTIGATOR: F. S. Owens
ABSTRACT: An intensive literature survey on elastomer stress relaxation revealed that previous investigators had been primarily concerned with the environmental effects of oxygen and temperature upon the rate of stress relaxation of elastomers rather than with the isolation of variables directly involved in the preparation of elastomeric vulcanizates. A systematic investigation of such variables as the milling time, vulcanization time, elongation rate, and the amounts of various compounding ingredients was therefore performed.

A medium acrylonitrile content butadiene-acrylonitrile elastomer was utilized exclusively as the base elastomer for the vulcanizates of this investigation. A compounding formula was selected as the "standard" stock, and the effects of the amounts of individual compounding ingredients were investigated individually through variations from this formula.

In addition to the investigation of the stress relaxation characteristics of butadiene-acrylonitrile vulcanizates per se, it was also the intent of this program to utilize the procedures developed in continuing studies of vulcanizates of other elastomers. This continuing study will utilize elastomers of high thermal stability and will involve measurements of stress relaxation at high temperatures.

ASD TN 61-34

February 1961

SUBJECT: ELASTOMERS IN SPACE ENVIRONMENTS, A
LITERATURE SEARCH
INVESTIGATOR: J. S. Delphenich
ABSTRACT: Flight of man into space is fast approaching

WADC TR 53-373 Sup 9

94

ASD TN 61-34 (Continued)

and the effect of space environments on elastomeric materials must be determined. This is a literature search of the effort that has been done in this area. Included are effects of ultraviolet radiation, high vacuum, and extreme temperatures on elastomeric materials.

ASD TR 61-76, Part I

September 1961

SUBJECT: ELASTOMERIC AND COMPLIANT MATERIALS FOR
CONTACT WITH LIQUID ROCKET FUELS AND
OXIDIZERS

INVESTIGATOR: J. Green, N. Levine

CONTRACT: AF33(616)-7227, Thiokol Chemical Corp.

ABSTRACT: Various techniques have been employed in efforts to provide elastomeric composites suitable for use in contact with hydrazine type fuels, nitrogen tetroxide, and chlorine tri-fluoride. Through the use of compounding studies and investigations of resinous and metallic coating systems several elastomeric materials have been recommended for field testing in contact with hydrazine type fuels and nitrogen tetroxide. A polyethylene coating has been applied to an elastomeric O-ring. The elastomer base has been protected from $\text{NO}_2 \rightleftharpoons \text{N}_2\text{O}_4$ for 24 hours by this coating.

Preliminary investigations have been initiated for development of elastomeric composites which are compatible with chlorine tri-fluoride.

ASD TR 61-84

September 1961

SUBJECT: ULTRAVIOLET RADIATION DEGRADATION OF
ELASTOMERS IN A HIGH VACUUM

INVESTIGATOR: C. D. Miller

CONTRACT: AF33(616)-7310, Armour Research Foundation

ABSTRACT: A series of twelve typical elastomers has been selected and exposed to short wave-length ultraviolet radiation and vacuum in a specially constructed chamber. Changes produced by irradiation have been followed by measurements of stress relaxation, weight loss, permanent set, and swelling. The samples are also examined for visible changes in the surface. Substantial differences in durability have been observed among these samples. For most of these elastomers, the primary reaction seems to be crosslinking, with only minor amounts of chain scission, and very little evolution of volatiles.

WADD TR 61-167

SUBJECT: MOLECULAR CONFORMATION AND ULTIMATE
PROPERTIES OF ELASTOMERS
INVESTIGATOR: M. Martin, N. Blesto, I. Piirma
CONTRACT: AF33(616)-6986, University of Akron
ABSTRACT: Films of butyl, pale crepe, polymethyl-
methacrylate, and styrene-butadiene polymers were cast from
"good" and "poor" solvents. These films were "quantitatively"
cross-linked and investigated in an effort to determine the
effect of the polymer-solvent interaction on the number of
physical chain entanglements as indicated and checked by
various measurements. Swelling, modulus, and sol measurements
showed a much higher proportion of chain entanglements in
films cast from "good" solvents than from "poor" solvents.

ASD TR 61-234

June 1961

SUBJECT: HANDBOOK OF DESIGN DATA ON ELASTOMERIC
MATERIALS USED IN AEROSPACE SYSTEMS
INVESTIGATOR: A. G. Pickett, M. M. Lemcoe
CONTRACT: AF33(616)-7093, Southwest Research Inst.
ABSTRACT: The objective of this handbook is to
provide aerospace weapons system design engineers with useful
data on the materials properties of elastomers. The sources
of this information are Department of Defense research re-
ports and the technical literature of engineering design and
elastomer technology. The elastomeric materials for which
data are presented are compounds of high polymers currently
available in the U.S.A. The properties considered are
original mechanical and physical properties and the changes
in these properties that result from aging and exposure to
environments of aerospace weapons systems. Elastomer com-
pounding is only briefly treated in this handbook because
it is intended for use by structural and mechanical engineers
rather than by rubber chemists and technologists. Elastomer
part design methods are not reviewed in this handbook because
they are the subjects of other Department of Defense reports
which this handbook is intended to complement. A selected
bibliography of technical literature on elastomers and
elastomeric parts is included to aid the handbook user who
needs further information on these topics.

SUBJECT: MECHANICALLY INITIATED REACTION OF ORGANIC MATERIALS IN MISSILE OXIDIZERS

INVESTIGATOR: R. L. Hauser, G. E. Sykes, W. F. Rumpel

CONTRACT: AF33(616)-7271, Martin Co.

ABSTRACT: This report presents the results of impact testing of 24 organic materials with liquid oxygen. In addition, nine of these same materials were tested with gaseous oxygen, and seven were tested with liquid oxygen tetroxide. Pure polymers, plasticizers, and anti-oxidants were studied and their threshold sensitivity levels and detonation energies were determined. Procedures and equations for calibrating impact testing machines were developed and used to calculate the rates of energy transfer into test materials. A full record of test procedures is included. In addition, 18 of the given materials in contact with liquid oxygen were subjected to shear forces with a modified Shell Four-Ball Wear Tester to determine whether reactions could be initiated by friction.

SUBJECT: COMPOUNDING OF HYDROCARBON ELASTOMERS FOR POTENTIAL HIGH TEMPERATURE APPLICATIONS

INVESTIGATOR: J.K. Sieron, K. Murray

ABSTRACT: Compounding studies on butyl, chlorobutyl, ethylene-propylene, and modified ethylene-propylene elastomers were conducted. The effect of the concentration of various types of carbon black on the mechanical properties of resin-cured butyl and chlorobutyl was studied at temperatures up to 450°F. Particle size and structure of the reinforcing carbon black were influential in imparting good mechanical properties to the vulcanizates at elevated temperatures. Stabilization systems for butyl and chlorobutyl vulcanizates are described. A system based on pentaerythritol and a phenylene diamine type stabilizer was particularly effective for chlorobutyl. Limited studies of modified ethylene-propylene elastomer showed that this new material has promise for applications requiring high strength at elevated temperature. Peroxide and "sulfur donor" type cures in conjunction with an amine stabilizer were especially promising. Nitrogen versus air heat-aging experiments indicated that oxidation rather than heat instability was responsible for degradation of ethylene-propylene vulcanizates at 400°F. Butyl and chlorobutyl vulcanizates exhibited little difference in tensile retention after the comparative heat-aging.

ASD TR 61-615

November 1961

SUBJECT: NEW MATERIALS FOR HIGH TEMPERATURE
PNEUMATIC TIRES

INVESTIGATOR: J. K. Sieron

CONTRACT: Data on HT-1, a promising new high temperature resistant fiber suitable for reinforcement on aircraft tires, are presented. The data indicate that HT-1 is far superior to other organic fibers used to reinforce aircraft tires in so far as thermal stability and radiation resistance are concerned. Compounding and physical property data on resin-cured butyl, the most promising current commercial elastomer for 450°F tires, are also given. The data show that butyl has adequate strength at temperatures up to 450°F, either unaged or after heat-soaking, for high temperature tire construction. The physical properties of new high strength silicone elastomers are given and indicate that this new class of elastomers may also be suitable for aircraft tire construction. Considerable data and tire-cord adhesive dip formulations for the HT-1 resin-cured butyl combination are presented. Data indicate that an adhesive dip based on a special resorcinol-formaldehyde resin provides improved adhesion of HT-1 tire-cord to resin-cured butyl rubber; however, much work remains to be done to gain adequate adhesion of elastomers to tire-cord at 400°F.

ASD TDR 62-248

February 1962

SUBJECT: SELF-SEALING OF AEROSPACE SHIPS BY THE
DOUBLE HOLLOW WALL CONCEPT

INVESTIGATOR: P. A. House

ABSTRACT: The presence of micrometeoroids in space poses a serious hazard to space vehicles. Puncture of such an object without some method of automatic self-sealing would result in a loss of life-supporting internal atmosphere. A method for providing this self-sealing capability is described. It consists of two fluid materials, a polymer and a cross-linking agent, separated by a thin membrane. When punctured the fluids mix, rapidly solidify, and seal the puncture.

ASD TDR 62-262

February 1962

SUBJECT: IMPROVING THE RESISTANCE OF INTEGRAL FUEL
TANK COATING MATERIAL TO FUEL ANTI-ICING
ADDITIVE

INVESTIGATOR: P. A. House

ABSTRACT: A major problem of jet aircraft is the

ASD TDR 62-262 (Continued)

formation of ice in the fuel system. It has been found that anti-icing additives will eliminate the ice but there is a compatibility problem between the additive and the coating material used in aircraft integral fuel tanks. This is a study of methods to make the coating more resistant to the additive. A method was chosen and subsequent evaluations performed to insure that all properties of the coating were within acceptable limits.

ASD TDR 62-286

March 1962

SUBJECT: COMPOSITE SEAL MATERIALS FOR EXTREME ENVIRONMENTS

INVESTIGATOR: R. E. Headrick

ABSTRACT: Unusual and extremely promising composite seal materials are critically discussed. These composite seal materials, resulting from an Air Force sponsored program with the Armour Research Foundation, are shown to be particularly promising for use in extreme aerospace environments. Evaluation of these materials, metal fiber skeletons impregnated with softer organic and inorganic materials, as seals at both static and dynamic conditions from liquid nitrogen temperatures to 1000°F and at pressures up to 5000 psi have yielded unsurpassed results. The metal fiber skeletons (stainless steel or molybdenum) provide the strength and resilience, and the impregnating materials (soft metals, soft metal alloys, or compounded polymeric materials) provide the sealing barrier. In some cases the impregnants have increased the resilience and lubricity of the composite. The limitations, potentials, and the availability of these materials are discussed.

COMPOSITES

WADC TR 59-338, Part III

March 1961

SUBJECT: COMPOSITE INORGANIC RESILIENT MATERIALS
INVESTIGATOR: L. L. Smith
CONTRACT: AF33(616)-5793, Armour Research Foundation
ABSTRACT: The principal objective of this research program is to investigate and develop composite materials suitable for use as static and dynamic seals at temperatures ranging from cryogenic to 1200°F and pressures up to 5000 psi.

Materials investigated may be grouped as follows: (1) Fibrous composites with pure metal impregnants. An example is stainless steel fiber skeletons impregnated with silver and/or indium with which promising results have been obtained at temperatures of 1000°F for reciprocating shaft seals and -424°F for static seals. (2) Fibrous composites with other impregnants. Included are metal skeletons filled with teflon, silicone, inorganic rubber, and various ceramic compositions. (3) Non-fibrous composites. These consist of ceramic composites containing talc or glass.

Included are studies of resilience, fiber orientation, friction, wear, static seals, reciprocating shaft seals, rotating shaft seals, and radiation effects.

WADC TR 59-749, Part II

June 1961

SUBJECT: COMPOSITE ELASTOMER-METAL O-RING SEALS
INVESTIGATOR: L. E. Doughty
CONTRACT: AF33(616)-7079, Chance Vought Corp.
ABSTRACT: This program is a continuation of the study initiated under Air Force Contract AF33(616)-6182. The objectives of this program were elastomer-metal composite O-ring seals with superior physical properties and evaluation of the performance of the prototype O-ring seals in adverse environments. A revised fabrication procedure, which improved the acceptability rate of composite O-ring seals, was developed to embed a metal spring concentrically in an elastomeric O-ring.

Two adhesives to bond the elastomer to the spring core were found suitable for use. Hydraulic dynamic cycling tests were performed at 4000 psi and temperatures up to 500°F. Performance of composite O-rings installed

WADC TR 59-749, Part II (Continued)

on a test piston without back-up rings was not satisfactory for practical use at elevated temperatures and pressures. Performance of composite O-ring seals used with back-up rings was comparable to that of conventional O-ring seals. Because of the severe degradation of elastomeric properties at high temperatures, the dynamic sealing performance of elastomeric O-ring seals, conventional or composite, appears to be dependent on back-up ring performance.

WADD TR 60-299

April 1961

SUBJECT: SYNTHESIS OF FIBER REINFORCED INORGANIC LAMINATES
INVESTIGATOR: J. H. Lauchner, D. G. Bennett
CONTRACT: AF33(616)-6283, Illinois University
ABSTRACT: Research was done on the matrix phase of glass fiber reinforced inorganic laminates. High temperature strength and stability of low elastic modulus matrix and high modulus fibers were objectives. Matrix glassy bonds, chloride, sulphate, spinel and phosphate bonds were studied with cleavable mineral and inorganic oxide type filler materials. Phosphate bonds, being best, were most studied. Phosphate bonded complex oxide bodies showing $AlPO_4$ and $FePO_4$ phases exhibited elastic moduli of about 0.3×10^6 psi and flexure strength up to 10,000 psi. Some bodies, as tested at 1000°F, deformed inelastically under loads of about 2000 psi. Such bodies were quite dense and moisture resistant but glass fiber protection from their corrosive effect is indicated.

WADD TR 60-697

May 1961

SUBJECT: STUDY OF THERMAL RADIATION WITHIN SOLIDS AND STUDY OF INTERNALLY ABLATING COMPOSITES
INVESTIGATOR: F. A. Vassalo, H. C. Caminitz, H. P. Kirchner
CONTRACT: AF33(616)-6886, Cornell Aeronautical Laboratory, Inc.
ABSTRACT: Work reported deals with high temperature materials. Phase I is a study of thermal radiation within solids. Its objective is to study the feasibility of developing new materials of composites to provide either reductions or increases in radiant heat transfer in solids. Equations governing heat transfer by thermal radiation within solids are derived and collected. The mechanisms for the reduction of heat transfer by thermal radiation in

WADD TR 60-697 (Continued)

solids are described and compared. Phase II concerns an analysis of composite materials so designed that high heat flux at one face will cause one phase to act as an energy absorbing material by melting, vaporizing or decomposing and thereby flowing out through the heated surface and in so doing, cool the other phase and maintain its dimensional stability. This combined action is referred to as internal ablation. Mathematical formulation of the basic processes is described and a numerical method is employed for solution.

ASD TR 60-697, Part II, Vol I

SUBJECT: INTERNALLY ABLATING COMPOSITES
INVESTIGATOR: F. A. Vassallo
CONTRACT: AF33(616)-6886, Cornell Aeronautical
Laboratory

ABSTRACT: The work reported concerns an analysis of composite materials so designed that high heat flux at one face will cause one phase to act as an energy absorbing material by melting, vaporizing, or decomposing and thereby flowing out through the heated surface and in so doing, cool the other phase and maintain its dimensional stability. This combined process is referred to as internal ablation.

The results of numerical analysis have been generalized and expressed by dimensionless groups whose relations are shown in chart form. The initially considered case of one-dimensional heat flow into a semi-infinite body has been followed by a similar treatment for the finite body. The curves prepared allow a computation of the direction of the two-phase body (limited by melting of the heated face of the matrix or by removal of all of the ablative phase) for many combinations of material and environmental characteristics. In a typical example given as an illustrative calculation, the duration of the two-phase body is several times greater than that of the non-porous matrix.

Experimental tests are described and their results generally confirm the results of the analysis.

WADD TR 60-697, Part II, Vol II

January 1962

SUBJECT: THERMAL RADIATION WITHIN SOLIDS
INVESTIGATOR: F. A. Vassallo, H. P. Kirchner
CONTRACT: AF33(616)-6886, Cornell Aeronautical
Laboratory

WADD TR 60-697, Part II, Vol II (Continued)

ABSTRACT: The study reported concerns the transmission of energy by radiation within solids. Considerations were limited to investigation of methods by which radiant transport might be increased. A major portion of the work deals with an analytical evaluation of a particular combination of materials in which dissipation of radiant energy transfer by means of ablation is utilized to increase the dimensional stability of the heated surface. The analysis serves to illustrate the general methods used in the study of the combined processes of heat conduction and radiant emission and reabsorption in the solid. It is shown that with the temperatures expected in future rocket engines radiant energy transport can provide a means by which material temperatures are maintained at tolerable levels.

WADD TR 60-791, Part I

August 1961

SUBJECT: HIGH TEMPERATURE RESINS, ANALYSIS OF PROCESS PARAMETERS, AND EVALUATION PROCEDURES FOR FILAMENT WOUND COMPOSITES.
PART I - HIGH TEMPERATURE RESINS

INVESTIGATOR: J. V. Kindall, F. Fiel, S. Susman, S. Yurenda

CONTRACT: AF33(616)-6737, Narmco Industries, Inc.

ABSTRACT: The main objective of this phase of the program was to develop a superior heat resistant filament winding resin, effective at 500°F and for short time exposure at 750°F. Hoop tension and interlaminar shear tests, carried out on a unidirectional circular glass fiber-resin composite (NOL type ring) were used as the chief means of resin screening and evaluation. Available heat stable epoxy, epoxy novolac, temperature resistant polyester, silicone-phenolic, and silicone resins were evaluated. Two satisfactory synthesized epoxy resins, vinyl resorcinol diglycidyl ether and allyl diglycidyl cyanurate were developed. Dow's epoxy novolac, DEN 438/MNA/DMP-30, resin system was selected as the best over-all temperature resistant filament winding resin. Hoop tensile strengths (ring stress) of 157,000 psi at room temperature, 125,000 psi at 500°F and 84,000 psi at 750°F were obtained.

ASD TR 61-128

August 1961

SUBJECT: THERMO-ELASTIC EQUATIONS FOR A SANDWICH PANEL UNDER ARBITRARY TEMPERATURE DISTRIBUTION, TRANSVERSE LOAD AND EDGE COMPRESSION

INVESTIGATOR: Ibrahim K. Ebcioğlu

ABSTRACT: The problem of designing sandwich panels for use under an arbitrary temperature distribution, transverse load, and edge compression has been extremely simplified. The core of the sandwich panel is assumed to be orthotropic and the faces may be of different thicknesses and materials. The general differential equations for a sandwich panel are obtained from the principles of mechanics, and corresponding boundary conditions are formulated from the principle of virtual displacements and variational calculus. A reduction from five differential equations for a sandwich panel to two independent systems of differential equations is accomplished by the suitable transformations of the independent variables. The final two systems of differential equations are solved simultaneously for an arbitrary temperature distribution, transverse load, and uniform edge compression with simply-supported boundary conditions. The analysis is greatly simplified by the use of the superposition principle. The theoretical solution is applied to a particular temperature distribution and center deflection in the sandwich panel plotted against loading parameter.

ASD TR 61-215

September 1961

SUBJECT: LITERATURE SURVEY ON FILAMENT-WOUND COMPOSITE STRUCTURES

INVESTIGATOR: J. A. Rolston

ABSTRACT: Filament Winding is the newest phase in the state-of-the-art of structural reinforced plastics. This report brings together abstracts of existing literature relating to filament winding.

ASD TR 61-300

June 1961

SUBJECT: THE PROPERTIES OF COMPRESSION MOLDED, GLASS FLAKE REINFORCED, RESIN COMPOSITES

INVESTIGATOR: A. J. Luirette

CONTRACT: AF33(616)-7195, Narmco Industries, Inc.

ABSTRACT: Four molding compositions using glass flake as reinforcement were selected for study by means of a series of screening tests. The physical, electrical and

mechanical properties of the compositions after compression molding into flat laminates were determined. Simple geometric shapes such as a hemisphere were compression molded from these compositions. The properties of the simple shapes were compared to the properties of the flat laminates. It was observed that movement of the resin-flake mixture in the molds resulted in very poor alignment of the flakes within the molding. This improper alignment resulted in significantly reduced mechanical properties. It was concluded that this problem is inherent to presently available glass flake reinforced molding compounds and will prohibit the satisfactory compression molding of everything but extremely simple shapes such as flat plates using available molding compounds and molding techniques.

ASD TR 61-338

May 1961

SUBJECT: FATIGUE TESTING OF HONEYCOMB CONSTRUCTIONS
INVESTIGATOR: H. Spector
CONTRACT: AF33(616)-7200, Northrop Corp.
ABSTRACT: Dynamic methods, developed by Northrop Corp., Norair Division, for determination of Young's modulus for honeycomb sandwich constructions, were successfully applied to obtain honeycomb core fatigue properties. S-N curves for extensional core fatigue were developed for representative samples of brazed, welded, and adhesive bonded honeycomb constructions. For tension - compression, the data shows that core density is a dominant parameter in core fatigue. There are indications that core thickness up to and including 1-inch has little effect upon the data; more investigation is necessary to determine the effect of thickness greater than 1-inch. Feasibility of obtaining core fatigue using a two specimen shear modulus approach was demonstrated, but more development is necessary in the design of testing systems to obtain dynamic balance and proper load levels.

ASD TDR 62-286

March 1962

SUBJECT: COMPOSITE SEAL MATERIALS FOR EXTREME ENVIRONMENTS
INVESTIGATOR: R. E. Headrick
ABSTRACT: Unusual and extremely promising composite seal materials are critically discussed. These composite seal materials, resulting from an Air Force sponsored program

with the Armour Research Foundation, are shown to be particularly promising for use in extreme aerospace environments. Evaluation of these materials, metal fiber skeletons impregnated with softer organic and inorganic materials, as seals at both static and dynamic conditions from liquid nitrogen temperatures to 1000°F and at pressures up to 5000 psi have yielded unsurpassed results. The metal fiber skeletons (stainless steel or molybdenum) provide the strength and resilience, and the impregnating materials (soft metals, soft metal alloys, or compounded polymeric materials) provide the sealing barrier. In some cases the impregnants have increased the resilience and lubricity of the composite. The limitations, potentials, and the availability of these materials are discussed.

EROSION

WADD TR 60-193

July 1961

SUBJECT: THERMAL EROSION OF ABLATIVE MATERIALS
INVESTIGATOR: D. L. Robbins, G. Epstein
CONTRACT: AF33(616)-6285, Aerojet-General Corp.
ABSTRACT: The thermal erosion resistance of 55 plastic nozzles was determined under rocket nozzle conditions. A 150-lb thrust, gaseous, hydrogen-oxygen rocket motor was used to investigate the behavior of specimens composed of a continuous-phase resin, usually reinforced with fibers of high-silica glass, alumino-silicate glass, graphite, or nylon and sometimes filled with ceramic or graphite particles. The experimental method provided information on the comparative thermal-erosion-resistant properties of the various test specimens.

PLASTICS

WADC TR 52-183, Sup 9

November 1961

SUBJECT: ANNUAL REPORT ON RESEARCH IN ANC-17
HANDBOOK "PLASTICS FOR FLIGHT VEHICLES"
INVESTIGATOR: D. G. Coleman
CONTRACT: D033(616)-61-06, U.S. Department of
Agriculture
ABSTRACT: Developments in the program of research
in plastics for flight vehicles conducted by the U.S. Forest
Products Laboratory during Fiscal Year 1961 are summarized.
In general, the approach has been to derive criteria mathe-
matically, and then to check by test. Two technical reports
were issued during the fiscal year and are abstracted.

WADC TR 59-600, Part II

April 1961

SUBJECT: MECHANISM OF REINFORCEMENT OF FIBER-
REINFORCED STRUCTURAL PLASTICS AND
COMPOSITES
INVESTIGATOR: J. S. Islinger, K. Gutfreund, I. M. Daniel,
A. J. Durelli
CONTRACT: AF33(616)-6987, Armour Research Foundation
ABSTRACT: This is a continuation of the research
effort undertaken to study the mechanism of reinforcement in
fiber-reinforced structural plastics and composites.
Analytical and experimental approaches include physical-
chemical investigations of fiber-resin interfaces, photo-
elastic investigations of composites, and studies of the
mechanical behavior of composites.

Data is presented for chemisorption
studies of the interactions of water with bare, silane-
treated, silane-pretreated, and polymer-reacted glass fibers
and of the reactions of alkenylsilane-treated E-glass with
styrene under simulated service conditions. Photo-elastically
determined shrinkage and other residual stresses around single
and groups of glass inclusions in cured polyester resins and
simulated composites are also presented. The mechanical
properties in flexure of parallel fiber-reinforced composites
with variations in reinforcement material and placement are
given, together with data resulting from studies of the
supposed orthotropic nature of the resin in fiber-reinforced
composites.

WADC TR 53-373 Sup 9

108

WADD TR 60-24

March 1961

SUBJECT: HIGH MODULUS, HIGH TEMPERATURE GLASS FIBERS FOR REINFORCED PLASTICS

INVESTIGATOR: G. R. Machlan, C. J. Stalego, R. L. Tiede, A. B. Isham, D. E. Caramante

CONTRACT: AF33(616)-5802, Owens-Corning Fiberglas Corp.

ABSTRACT: An exploratory research investigation to determine the feasibility of producing glass fibers which retain useful properties at temperatures up to 2500°F, resulted in the development of fibers which had a strength of 110,000 psi at 1800°F, an elastic modulus of 12.8×10^6 psi at room temperature, and a specific gravity of 2.53. A program to improve the strength properties of laminates made from the high modulus glass developed in Suppl 1 of the contract resulted in laminates which had wet strengths at least equivalent to similar "E" glass laminates and an increase of 34 percent in wet flexural modulus. A program to develop unidirectional, non-woven, preimpregnated reinforcement which would produce laminates having very high strength yielded laminates which had a dry flexural strength of 265,000 psi and a dry flexural modulus of 7.81×10^6 psi.

WADD TR 60-109, Part II

August 1961

SUBJECT: INSULATION MATERIALS FOR SOLID PROPELLANT ROCKET MOTORS

INVESTIGATOR: J. Batchelor, N. Vasileff, S. McCormick, E. Olcott

CONTRACT: AF33(616)-6831, Atlantic Research Corp.

ABSTRACT: Research on ablative insulation materials intended for use in high temperature environments of rocket exhausts was devoted to (a) establishment of realistic performance criteria for the improvement of ablative insulation composites, (b) relationships between laboratory characterization and small scale motor firings for investigation of candidate materials, and (c) synthesis and formulation of new, high performance ablative insulation materials. Performance criteria established for the investigation of rocket insulation materials included: material erosion rate, charring rate, uniformity of ablation, and similar considerations. Surface chars produced on insulation materials exposed to high temperatures were investigated. Several new flexible insulation materials, based primarily on polyurethane and modified epoxy resins were formulated.

WADD TR 60-110, Part II

March 1961

SUBJECT: THERMAL PROTECTIVE SURFACES FOR STRUCTURAL PLASTICS
INVESTIGATOR: J. W. Vogan
CONTRACT: AF33(616)-6393, Bendix Products Div.
ABSTRACT: A study was made of protective surfaces for plastics in high temperature and velocity gas streams. Testing was conducted on a solid fuel rocket with a gas temperature of approximately 6800°F. Results showed that the lightweight zirconia ceramics in combination with a plasma sprayed coating provided the best protective for the plastic substrate.

WADD TN 60-286

January 1962

SUBJECT: PROPERTIES OF THERMALLY DEGRADED ABLATIVE PLASTICS
INVESTIGATOR: H. S. Schwartz, D. L. Schmidt, S. A. Marolo, D. F. Starks
ABSTRACT: A comprehensive investigation was performed to determine the physical, chemical, and mechanical properties of thermally degraded plastic ablation materials. Several new experimental techniques were developed and some existing techniques were modified to obtain the desired information. Deductions from experimental results provide new information on ablation phenomena.

WADD TR 60-647

August 1961

SUBJECT: THERMAL IRRADIATION OF PLASTIC MATERIALS
INVESTIGATOR: H. S. Schwartz, R. W. Farmer
ABSTRACT: Degradation and short time rupture of several reinforced plastics and structural adhesives were investigated during intense radiant heating. Thermal response was found to be a function of certain materials properties, stress conditions and environmental parameters.

Plastic specimens were preloaded in tension and irradiated in an arc-imaging furnace. Time to rupture was found to be a function of: resin and reinforcement type, spectral characteristics and thickness of the material, angle and magnitude of the applied static stress, and radiant flux density. Rupture times ranging from 0.5 to 45 seconds were observed for applied stresses of 20 to 70 percent of the ultimate room temperature strength and radiant flux densities of 1 to 25 Cal/cm²-sec.

WADD TR 60-647 (Continued)

An exploratory study was made on an adhesive bonded clad aluminum lap joint loaded in shear and irradiated at 25 Cal/cm²-sec. Rupture times of 1 to 10 seconds were obtained for static loads of 30 to 70 percent of ultimate room temperature strength.

WADD TR 60-697

May 1961

SUBJECT: STUDY OF THERMAL RADIATION WITHIN SOLIDS AND STUDY OF INTERNALLY ABLATING COMPOSITES

INVESTIGATOR: F. A. Vassallo, H. C. Caminitz, H. P. Kirchner

CONTRACT: AF33(616)-6886, Cornell Aeronautical Laboratory, Inc.

ABSTRACT: Work reported deals with high temperature materials. Phase 1 is a study of thermal radiation within solids. Its objective is to study the feasibility of developing new materials of composites to provide either reductions or increases in radiant heat transfer in solids. Equations governing heat transfer by thermal radiation within solids are derived and collected. The mechanisms for the reduction of heat transfer by thermal radiation in solids are described and compared. Phase 2 concerns an analysis of composite materials so designed that high heat flux at one face will cause one phase to act as an energy absorbing material by melting, vaporizing or decomposing and thereby flowing out through the heated surface and in so doing cool the other phase and maintain its dimensional stability. This combined action is referred to as internal ablation.

Mathematical formulation of the basic processes is described and a numerical method is employed for solution.

ASD TR 60-697, Part II, Vol I

January 1962

SUBJECT: INTERNALLY ABLATING COMPOSITES

INVESTIGATOR: F. A. Vassallo

CONTRACT: AF33(616)-6886, Cornell Aeronautical Laboratory, Inc.

ABSTRACT: The work reported concerns an analysis of composite materials so designed that high heat flux at one face will cause one phase to act as an energy absorbing

material by melting, vaporizing, or decomposing and thereby flowing out through the heated surface and in so doing, cool the other phase and maintain its dimensional stability. This combined process is referred to as internal ablation.

The results of numerical analysis have been generalized and expressed by dimensionless groups whose relations are shown in chart form. The initially considered case of one-dimensional heat flow into a semi-infinite body has been followed by a similar treatment for the finite body. The curves prepared allow a computation of the duration of the two-phase body (limited by melting of the heated face of the matrix or by removal of all of the ablative phase) for many combinations of material and environmental characteristics. In a typical example given as an illustrative calculation, the duration of the two-phase body is several times greater than that of the non-porous matrix.

Experimental tests are described and their results generally confirm the results of the analysis.

WADD TR 60-697, Part II, Vol II

January 1962

SUBJECT: THERMAL RADIATION WITHIN SOLIDS
INVESTIGATOR: F. A. Vassallo, H. P. Kirchner
CONTRACT: AF33(616)-6886, Cornell Aeronautical
Laboratory, Inc.

ABSTRACT: The study reported concerns the transmission of energy by radiation within solids. Considerations were limited to investigation of methods by which radiant transport might be increased. A major portion of the work deals with an analytical evaluation of a particular combination of materials in which dissipation of radiant energy transfer by means of ablation is utilized to increase the dimensional stability of the heated surface. The analysis serves to illustrate the general methods used in the study of the combined processes of heat conduction and radiant emission and reabsorption in the solid. It is shown that with the temperatures expected in future rocket engines radiant energy transport can provide a means by which material temperatures are maintained at tolerable levels.

WADD TR 60-735

September 1961

SUBJECT: HIGH MODULUS GLASS FIBERS FOR REINFORCED PLASTICS
INVESTIGATOR: G. P. Peterson
CONTRACT: AF33(616)-5500
ABSTRACT: This report is a compilation of papers and a general discussion presented at the Aeronautical Systems Division - University of Dayton joint meeting on High Modulus Fibers for Reinforced Plastics, 12 October 1960, Miami Hotel, Dayton, Ohio. The papers, for the most part, reviewed work completed or in process under Directorate of Materials and Processes research and development programs.

WADD TR 60-797

December 1960

SUBJECT: LABORATORY TECHNIQUES FOR DETERMINING THE HIGH TEMPERATURE STRUCTURAL BEHAVIOR OF REINFORCED PLASTICS
INVESTIGATOR: R. J. McBride
CONTRACT: AF33(616)-7004, University of Dayton
ABSTRACT: This report presents time-temperature flexural data obtained on reinforced plastics to establish standard elevated temperature exposure and test procedures. This program included the investigation of: time-temperature properties, the most satisfactory short time exposure period, and cycling type exposure which included simulation of service conditions. Based on the mechanical property data obtained utilizing typical reinforced plastic materials it was determined that the most important criteria involved in elevated temperature determination of mechanical properties were time at temperature and cyclic exposures. Optimum methods and exposure procedures are recommended for short and long time elevated temperature structural studies of reinforced plastic materials.

WADD TR 60-804

February 1961

SUBJECT: TENSILE AND COMPRESSIVE STRENGTH OF REINFORCED PLASTIC LAMINATES AFTER RAPID HEATING
INVESTIGATOR: K. H. Boller
CONTRACT: D033(616)-58-1, Forest Products Laboratory
ABSTRACT: The effect of rapidly heating the specimens with plate heaters or by oven is described. Tension and compression tests were made on three plastic laminates after exposure to temperatures ranging from room temperature to

WADC TR 53-373 Sup 9

113

WADD TR 60-804 (Continued)

1000°F and soak periods ranging from about 2 minutes to 1,000 hours. Results show that strength of laminates decreases with heat. Degree of degradation is a function of the kind of material and the temperature and period of exposure; hence, each material should be judged separately.

WADD TR 60-856

August 1961

SUBJECT: MICROSTRUCTURE OF ABLATIVE PLASTIC CHARS
INVESTIGATOR: S. A. Marolo
ABSTRACT: Techniques were derived for visual study of the microstructure of ablative plastic chars under high magnification.

The procedures consisted of (1) vacuum impregnation of the char with a thermosetting resin (2) curing the resin to rigidize the char (3) surface polishing the impregnated char and (4) obtaining magnified photographs of the char cross-section.

Information obtained from the photographs included (1) cell shape and size (2) wall thickness of cells and (3) carbon deposition and growth phenomena. This information was correlated with additional research data from other investigators on ablation of plastics to obtain a better understanding of the ablation process.

WADD TR 60-862

August 1961

SUBJECT: ABLATIVE PLASTICS FOR RE-ENTRY THERMAL PROTECTION
INVESTIGATOR: D. L. Schmidt
ABSTRACT: Ablation - a phenomenal heat and mass transfer process in which extreme environmental temperatures and aerodynamic heating may be accommodated with ease. An expository analysis of this process is presented as it relates to plastic materials. It is shown that ablative performance depends critically upon both materials and environmental variables. Each of these basic variables are analyzed and reviewed in detail.

Material research re-entry vehicles for flight testing of ablative thermal protection systems are discussed. Future re-entry environments, and associated material requirements are also reported.

WADC TR 53-373 Sup 9

114

WADD TN 61-48

November 1961

SUBJECT: ABLATIVE THERMAL PROTECTION FOR AEROSPACE VEHICLES
INVESTIGATOR: D. L. Schmidt
ABSTRACT: Thermal protection of hypersonic atmospheric vehicles with various cooling techniques has lead to the solution of one of the most challenging and difficult problems of aerospace flight. Various heat protective systems are now available for successful accommodation of the searing heat and dynamic forces associated with entry and flight in a planetary atmosphere. Of these protective systems, ablative cooling offers the greatest potential and thus has received the most attention.

This publication presents information on current and future aero-thermochemical flight regimes of aerospace vehicles. Practical methods are discussed for alleviating extreme aerodynamic heating, with emphasis on ablative cooling and materials. Plastics are shown to possess the greatest inherent capability among existing ablative materials. New environments encountered in the future will place more critical demands upon ablative materials. These environmental trends and associated material requirements are given in detail.

ASD TN 61-50

June 1961

SUBJECT: OPTIMUM FILAMENT WOUND COMPOSITES
INVESTIGATOR: G. P. Peterson
ABSTRACT: A review of the present Air Force program to obtain optimum filament wound composites is presented. The basic philosophy involves research in four major areas: 1) reinforcement and finishes, 2) matrix materials, 3) winding patterns and 4) fabrication and processing techniques. The latter two are directly related to filament winding and are treated as an integral unit in this review. The present state-of-the-art in each of these areas is reviewed and the significance and contribution of the Materials Central research effort toward providing advanced capabilities is discussed.

ASD TR 61-204

September 1961

SUBJECT: MECHANISM OF ABLATION OF CHAR-FORMING ABLATIVE PLASTICS
INVESTIGATOR: C. E. Lapple, A. P. Brady, D. L. Chamberlain, Jr.

WADC TR 53-373 Sup 9

115

ASD TR 61-204 (Continued)

CONTRACT: AF33(616)-7362, Stanford Research Inst.
ABSTRACT: A char-forming organic polymer was chosen as a model ablating material. Equations were developed and evaluated to show the relationship between ablation rate and char properties for various aerodynamic conditions, and for various physical and chemical properties of the polymer and its thermal degradation products. A novel char-simulating reactor was developed for measuring properties of decomposition products, in situ and without cooling, resulting from the passage of hydrocarbon gases through a synthetic char layer. Preliminary work established the feasibility of the technique, with temperature and pressure measurement instruments responding to a 0.1- to 1.0- second pulsed-flow. Operating temperatures of 3000°K have been obtained. Although feasibility of the technique has been established, a demonstration of the full capability of the technique has not been possible during the short period of operation.

ASD TR 61-304

January 1962

SUBJECT: EFFECTS OF HYPERTHERMAL CONDITIONS ON PLASTIC ABLATION MATERIALS
INVESTIGATOR: R. D. Buhler, D. Christensen, S. Grindle
CONTRACT: AF33(616)-7110, Plasmadyne Corp.
ABSTRACT: Comparative ablation behavior of plastic and composite ablation materials: 1) nylon fabric 2) silica fabric and 3) graphite cloth was evaluated in a hyperthermal plasma arc facility. The effect of various re-entry flight conditions was investigated in addition to the effect of dynamic pressure at a constant cold-wall heat flux on the ablation behavior of these materials. The importance of grain structure on the reliability of ablation behavior and measurements was evident in these tests, indicating a need for gross "screening" tests on the various types of materials prior to a final detailed evaluation in a plasma arc. Materials which "structurally" fail when exposed to the extreme environment imposed by a hyperthermal supersonic jet, are difficult to evaluate.

ASD TR 61-439, Part I

August 1961

SUBJECT: CRITERIA FOR PLASTIC ABLATION MATERIALS AS FUNCTIONS OF ENVIRONMENTAL PARAMETERS
PART I - RESULTS OF ANALYTICAL STUDIES
INVESTIGATOR: B. McFarland, P. Joerg, M. Taft

WADC TR 53-373 Sup 9

116

ASD TR 61-439, Part I (Continued)

CONTRACT: AF33(616)-7401, Aerojet-General Corp.
ABSTRACT: A mathematical model for the ablation of heterogeneous materials is developed and compared with experimental data for phenolic-asbestos, phenolic-nylon, phenolic-Refrasil and melamine-glass cloth laminates. Good agreement between theory and experiment was obtained for both transient and steady-state data. The pertinent physical parameters are discussed in terms of their influence on insulation requirements, and general requirements for an optimum insulation system are developed. Error analyses are presented to evaluate the effects of body geometry, variable ablation rate, and conductive substrates on heat penetration into the unchanged plastic.

ASD TR 61-439, Part II

August 1961

SUBJECT: CRITERIA FOR PLASTIC ABLATION MATERIALS AS FUNCTIONS OF ENVIRONMENTAL PARAMETERS PART II. Ablation Information Abstracts
INVESTIGATOR: V. M. Vila, D. Nilson
CONTRACT: AF33(616)-7401, Aerojet-General Corp.
ABSTRACT: Reports presenting ablation information and related technology have been compiled and abstracted. Ablation information abstracts of the various reports with author, corporate, and subject indexes are presented. The abstracted reports presented are believed to represent 35 percent of the available literature on the subject of ablation and related technology. Abstracts of 243 reports are presented.

ASD TR 61-482

March 1962

SUBJECT: STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES (CTL 37-9X Resin and 181-A1100 Glass Fabric)
CONTRACT: D033(616)-58-1 and 61-06, Forest Products Laboratory
INVESTIGATOR: K. H. Boller
ABSTRACT: Strength properties at elevated temperatures are presented for a phenyl-silane laminate made of CTL 37-9X resin and 181-A1100 glass fabric. In general, strength properties decrease with increases in temperature. From the curves and data, effects of temperatures between 800° and 1,000°F and exposure periods between 0.05 hour and 1,000 hours on individual strength properties may be judged and interpreted separately. Appendix presents the test methods used to obtain data.

ASD TR 61-517

January 1962

SUBJECT: LABORATORY TECHNIQUES FOR STUDYING
THERMALLY ABLATIVE PLASTICS
INVESTIGATOR: H. S. Schwartz
ABSTRACT: The utilization of plastic ablative-
insulative materials in re-entry and propulsion environments
and the factors governing their performance are briefly
described.

The pertinent material "response characteristics" are separated into three major categories: thermal, chemical, and physical-mechanical. Laboratory procedures are described. They include techniques for determining surface temperature and emissivity in hot gas streams; molecular weight and chemical identity of gaseous pyrolysis products; weight fractions of gaseous and residual solids from pyrolyzed plastics; heat of decomposition of plastics; apparent thermal diffusivity and thermal insulating characteristics; mechanical properties and cell structures and other physical characteristics of residual chars from pyrolyzed plastics.

ASD TR 61-650

February 1962

SUBJECT: ABLATION OF PLASTICS
ABSTRACT: This technical report is an authoritative presentation of the inter-disciplinary subject of materials ablation, particularly as it relates to thermally protective plastics and composites. It treats in detail many aspects of plastic ablation such as materials requirements, historical development, unique materials advantages and limitations, basic insights into the ablation phenomenon, a simplified theory, materials performance criteria, evaluation techniques, material-environment interactions, thermo-structural design, and typical applications.

The ultimate goal of this report is to convert the "art" of plastic ablation into an organized "engineering science". It is intended that the basic information presented will provide a basis for accelerated advancements in this new material engineering field.

FIBROUS MATERIALS

WADD TN 60-298, OTS Release

August 1961

SUBJECT: ELEVATED TEMPERATURE BEHAVIOR OF FIBERS
INVESTIGATOR: S. Schulman
ABSTRACT: The primary purpose of this research program is to study the behavior of fibers under conditions of high temperatures. The first phase covers the installation and calibration of equipment and establishment of operating techniques.

WADD TN 60-299

June 1961

SUBJECT: THERMAL AND GAMMA RADIATION BEHAVIOR OF A NEW HIGH TEMPERATURE ORGANIC FIBER
INVESTIGATOR: Capt C. Little
ABSTRACT: This research study involved a new temperature resistant experimental organic fiber known as HT-1, whose chemical structure is a departure from the conventional polyamide and polyesters. The behavior of this fiber during and after exposure to temperatures up to 650°F alone and in conjunction with gamma radiation indicates that a major breakthrough in organic fiber technology has been achieved. Specifically, exposure to ionized radiation for all practical purposes does not affect the tensile or elongation of HT-1 yarn. Tensile retention at 500°F is increased from 84.8% to 91.6% after combined thermal-ionized radiation exposure and at 600°F tensile retention is increased from 52.2% to 78.2%. The superior behavior of HT-1 can be exploited and affect the greatest overall advance in temperature resistant fiberology through utilization in aircraft and personnel deceleration systems; aircraft tires, ducting, fuel diaphragms and expulsion bags; reinforcement for ablating plastics for re-entry heat shields and other hyperthermal applications.

WADD TR 60-311

December 1960

SUBJECT: RESEARCH ON MATERIALS FOR USE IN A FULL PRESSURE SUIT
INVESTIGATOR: D. M. Mellen, S. Backer, B. J. Park
CONTRACT: AF33(616)-5974, Mass. Inst. of Tech.
ABSTRACT: This report presents the results of analytical and experimental studies directed towards design of a textile structure capable of use in a full pressure

WADD TR 60-311 (Continued)

suit. The properties of the fiber have been considered, but in a role secondary to that of the geometric mechanics of the structure. A pressurized testing device has been designed and used in measuring the bending and torsional rigidity of numerous sample-fabric cylinders.

WADD TR 60-327, OTS Release

February 1961

SUBJECT: TEXTILE FIBERS IN HIGH TEMPERATURE APPLICATIONS
INVESTIGATOR: R. G. Cheathan, M. E. Shank, S. Backer
CONTRACT: AF33(616)-5881, Mass. Inst. of Tech.
ABSTRACT: This report presents a survey of literature in the fields of metals, polymers and glasses undertaken to ascertain the availability of fibrous materials suitable for high temperature applications in re-entry parachutes.

Wherever possible, mechanical behavior of the material's fiber form was considered at the temperature level of 1500°F. But in many instances, only bulk behavior of the material in question has been reported with an indication of its fabricating problems. It may be expected that continued work on such materials will, in a few years, enlarge the stable of fiber candidates for high temperature textile performance. Meanwhile, there is at hand, a small selection of stable, high temperature metal fibers capable of meeting the re-entry parachute requirements although with some restrictions in weight, cost, and "textile performance".

WADD TR 60-511, Part II

February 1962

SUBJECT: INVESTIGATION OF THE HIGH SPEED IMPACT BEHAVIOR OF FIBROUS MATERIALS. PART II. Impact Characteristics of Parachute Materials
INVESTIGATOR: R. J. Coskren, C. C. Chu
CONTRACT: AF33(616)-7627, Fabric Research Labs, Inc.
ABSTRACT: A technique has been developed which effectively characterizes the impact behavior of high strength parachute components. The present equipment will measure force and entension build-up in a webbing during impact. Further refinements in the test equipment are required to make precision measurement practical. One modification is

WADC TR 53-373 Sup 9

120

WADD TR 60-511, Part II (Continued)

the use of high-speed motion pictures rather than multiple-exposure stills.

Our results have shown that slow-speed (Instron) tensile behavior is not always indicative of impact response. Certain configurations are more efficient than others.

Further structural parametric studies of HT-1 materials in parachute components is recommended since results obtained during this program indicates a significant loss in energy absorption capability of the HT-1 webbings studied at impact velocities in excess of 500 feet per second.

WADD TR 60-584, Part II, OTS Release

October 1961

SUBJECT: HANDBOOK OF FIBROUS MATERIALS
INVESTIGATOR: E. R. Kaswell
CONTRACT: AF33(616)-7504, McGraw-Hill Book Co.
ABSTRACT: This report contains the summarized and consolidated information extracted from ASD Technical Reports covering several phases of fibrous materials research. The information is arranged to make the results of these reports more readily available and useful to decelerator designers and others interested in the fibrous materials phase of Air Force research.

The report is divided into sections covering the pertinent facets of fibrous material information. Fairly complete information is supplied on various phases of ageing properties; basic design data; friction, abrasion and weather resistance; impact loading; porosity and air permeability; sewability; radiation properties; and aerodynamic heating of different yarns, cords, webbings, and fabrics.

WADD TR 60-588, OTS Release

June 1961

SUBJECT: SOME EFFECTS OF COMPRESSION AND HEAT ON DECELERATOR MATERIALS
INVESTIGATOR: N. J. Abbott
CONTRACT: AF33(616)-6738, Fabric Research Labs, Inc.
ABSTRACT: The degradation of some nylon parachute materials, as well as similar materials made from Dacron, glass, an unnamed organic fiber, and stainless steel, when subjected to varying conditions of temperature, pressure,

WADD TR 60-588 (Continued)

and time was studied. Temperatures up to 1000°F, pressures up to 250 psi, and times up to 72 hours were used. The amount of degradation for most of the materials was found to be consistent with that reported by other authors who did not study the influence of pressure. Pressure was less important than temperature or time, but it did tend to decrease degradation somewhat because of the reduction in the amount of oxygen contained within the structure. All of the materials, with the exception of the unnamed organic fiber and steel, showed marked stiffening at 350°F, due primarily to inter-fiber sticking. Examination of a pressure-packed parachute which had been heated to 380°F showed serious stiffening and setting in the folded configuration, making it very doubtful that successful deployment could have been achieved.

WADD TR 60-670, Part I, OTS Release

December 1960

SUBJECT: INSTANTANEOUS LOCAL TEMPERATURES OF AERO-DYNAMIC DECELERATORS, PART I. Methods of Predicting
INVESTIGATOR: G. Engholm, R. J. Baschiere, R. A. Bambenek
CONTRACT: AF33(616)-6673, American Machine & Foundry Co.
ABSTRACT: A numerical technique is presented for predicting the temperatures developed in aerodynamic decelerators at high speeds. The predictions are obtained both by a simple method to estimate local equilibrium temperatures and by a more complex method to estimate instantaneous, local temperature profiles. Sample calculations are included. A new numerical technique is described for predicting one dimensional thermal diffusion. This technique permits the use of larger time increments and therefore fewer calculations than necessary with other numerical techniques.

WADD TR 60-670, Part II, OTS Release

February 1961

SUBJECT: INSTANTANEOUS LOCAL TEMPERATURES OF AERO-DYNAMIC DECELERATORS PART II. Thermal Properties
INVESTIGATOR: G. Enghelm, S. J. Lis, R. A. Bambenek
CONTRACT: AF33(616)-6673, American Machine & Foundry Co.
ABSTRACT: Measurements were made of the thermal conductivity of the following candidate parachute fabrics: nylon, stainless steel, glass, graphite, and AF-1 (an

WADD TR 60-670, Part II (Continued)

organic fiber). Data was obtained with a Cenco-Fitch device in which the effects of compressive load was established. A similarity relation derived from dimensional analysis was applied to this data and resulted in a satisfactory correlation.

A new apparatus was developed in order to determine fabric conductivity under varying conditions of temperature (212 to 600°F), ambient pressure (15 to 0.048 psia), bi-axial tension (0 to 90 ppi), and compression (1 to 10 psi). Initial data collected using the apparatus demonstrated its capabilities and analysis of the data indicate the possibility of further dimensionless correlations may be negligible. Considerably more detailed data will have to be obtained to establish repeatability and verification of these hypotheses.

WADD TR 60-897

July 1961

SUBJECT: THE THEORETICAL BEHAVIOR OF KNITTED
FABRIC SUBJECTED TO BIAXIAL STRESSES

INVESTIGATOR: P. Popper

ABSTRACT: In this report, the theoretical mechanical behavior of a plain knitted fabric subjected to biaxial stresses is derived. An approximate mathematical model of the fabric structure has been established from which the stress vs. fabric-geometry and stress vs. strain relationships have been determined. The work was done by considering only the properties of the fabric structure, completely independent of the fiber properties. The results of this report may be used for such applications as predicting the performance of a plain knitted fabric in situations where it will be stressed biaxially.

ASD TR 61-6, OTS Release

June 1961

SUBJECT: DEVELOPMENT OF A PERMANENTLY FIRE-RESISTANT COTTON FABRIC BY REACTION WITH ORGANOBORON COMPOUNDS

INVESTIGATOR: R. W. Liggett, G. Bosmajian

CONTRACT: AF33(616)-6941, Southern Research Inst.

ABSTRACT: In the course of this study, approximately 100 preparations of organoboron compounds were carried out. Of these, about 50 compounds were evaluated as durable, flame-resistant finishes. Initial flame resistance was obtained

ASD TR 61-6 (Continued)

when desized and scoured cotton fabric was treated with the reaction product of boron trifluoride and aniline; when aminocellulose was treated with B-trichloroborazole; and when alkali cellulose was treated with some organo-boron halides. None of the treated fabrics were flame resistant after laundering. Organoboron compounds do not offer promise for the production of durable, flame-resistant effects on cotton fabrics.

ASD TN 61-59

August 1961

SUBJECT: FIBROUS MATERIALS FOR DECELERATORS AND STRUCTURES

INVESTIGATOR: J. C. McGrath

ABSTRACT: Decelerators of all types are normally over designed to insure reliable operation of the system. This over-designing is in the design of the system and also in the fibrous materials going into it, resulting in a system 10 to 15% stronger and heavier than is actually required. The designer of a decelerator system uses a pre-determined factor of safety which utilizes the minimum strength of a material (as specified in the applicable fibrous material specification) rather than over strength and with additional bulk to be packed into the decelerator storage compartment. In order to help alleviate this situation, a series of webbings were designed with specified strength as the main requirement. This was accomplished for six types of webbing ranging from minimum strength of 3000 lbs. to 10,000 lbs. The data obtained from these six acceptable types of webbing will be used as the basis for a new specification.

ASD TR 61-134, OTS Release

November 1961

SUBJECT: INVESTIGATION OF TECHNIQUES AND MATERIALS FOR THE FORMATION OF HIGH TEMPERATURE (1500°F) INORGANIC FIBER

INVESTIGATOR: R. B. Ellis

CONTRACT: AF33(616)-6172, Southern Research Inst.

ABSTRACT: The object of this project is to produce a high temperature (1500°F) inorganic fiber by forming the fiber with an organic matrix at a low temperature and firing this form to remove the organic part. The feasibility of the technique has been demonstrated on a laboratory scale by continuously firing an organically-bonded fiber to an entirely

WADC TR 53-373 Sup 9

124

ASD TR 61-134 (Continued)

inorganic form. The copolymer Acrilan is employed as the low-temperature fiber former and matrix. The inorganic materials consist of powdered silica or kaolin and low-melting frits. The service temperature of Acrilan has been extended to overlap the temperature at which a low-melting inorganic material softens enough to bond. Fiber strength in the transition range was developed by heat treating composite fiber containing zinc chloride at 200°C. and by selecting appropriate frits.

An entirely inorganic fiber 75 microns in diameter was fired continuously from a composite fiber composed of 25% Acrilan and 75% inorganic materials.

Information has been developed to permit selection of compositions and conditions allowing the formation of fibers.

ASD TR 61-192, OTS Release

June 1961

SUBJECT: A THEORETICAL AND EXPERIMENTAL STUDY OF
GAS FLOW THROUGH CLOTH OVER A RANGE OF
PRESSURES AND TEMPERATURES

INVESTIGATOR: F. O. Smetana

CONTRACT: AF33(616)-7102, University of Southern
California

ABSTRACT: Measurements were made of the permeability of five fabrics at downstream pressures from sea level to 150,000', pressure drops across the samples of 1 mm Hg to 900 mm Hg, and stagnation temperatures from 300°K to 930°K. In addition to this basic information, the investigation sought to provide a means of predicting high altitude results from those at sea level. It was found that: (a) the geometry of the test apparatus can have a marked influence on the results, (b) the major elastic effects on permeability arise from the change in fabric pore inclination with load rather than through simple extension of the yarn itself, (c) viscous effects are present for all except the very highest pressures, (d) rarefaction effects appear at altitudes above about 60,000', (e) the most satisfactory model for explaining the results appears to be one likening the flow to that between two non-interacting cylinders, and (f) as long as the fabric retains its elasticity and does not take a permanent set, temperature changes affect the permeability only insofar as the air density is changed. Some of these findings have not as yet been described quantitatively; for this reason, a satisfactory prediction procedure is not yet available.

ASD TR 61-227

October 1961

SUBJECT: FLAME-RESISTANT PARACHUTE PACK MATERIAL
INVESTIGATOR: R. F. Schwenker, Jr., R. K. Zuccarello
CONTRACT: AF33(616)-7009, Textile Research Inst.
ABSTRACT: This work involved the problem of obtaining a flame-resistant parachute pack material that would offer effective thermal protection to the parachute canopy contained therein. During the course of the work, a special apparatus and test method were developed to evaluate fabric heat transmission. Cotton duck fabrics were used as base materials and samples were subjected to (1) chemical modification and (2) resin treatments to improve fabric thermal characteristics. All treatments imparted excellent flame and glow resistance but did not significantly reduce heat transmission through the fabric.

ASD TR 61-518, OTS Release

December 1961

SUBJECT: DEVELOPMENT OF SHADE STANDARD AND TOLERANCES FOR CLOTH, COTTON, WIND-RESISTANT SATEEN, SHADE BLUE 1517
INVESTIGATOR: A. M. Herron
CONTRACT: AF33(616)-7907, Georgia Institute of Technology
ABSTRACT: Shade standards and tolerances were developed for combed cotton sateen, MIL-C-557D, in shade Blue 1517. Alternative dye formulations were developed by laboratory dyeings made in a fashion to simulate commercial continuous dye practice. Visual and instrumental characterization of the standard and tolerances was accomplished. A multipurpose reflectometer was used for instrumental characterization of the dyed materials.

The shade standard was produced in a commercial finishing plant using a continuous dyeing system with minor shade correction using a batch method.

The tolerance spacing was determined by visual means and the shade difference as detected by the instrument was then related to the selected shade standard. Although a measure of correlation was obtained, a number of eccentricities were encountered. The report discusses some reasons for these deviations.

ASD TR 61-560

November 1961

SUBJECT: THE MEASUREMENT OF ULTRAVIOLET REFLECTANCE
AS A CRITERION OF NYLON FABRIC DETERIORA-
TION

INVESTIGATOR: C. C. Chu, R. A. Kenney, E. R. Kaswell,
E. J. Stavrakas

CONTRACT: AF33(616)-7149, Fabric Research Labs, Inc.

ABSTRACT: A study was conducted to determine the
feasibility of utilizing changes in the ultraviolet reflectance
of nylon fabrics as a non-destructive measure of their de-
terioration when exposed to sunlight and to heat. Twenty-
four nylon parachute fabrics consisting of three weight classes
and eight finishing treatments on each were evaluated. Each
fabric sample was subjected to a variety of accelerated aging
treatments which included outdoor exposures in Massachusetts
and Arizona, and laboratory exposures in a Weatherometer and
in an oven. Breaking strength and ultraviolet reflectance
by use of a spectrophotometer were determined after each ex-
posure station. The correlation between these two parameters
was found to be in the 85% range; however, the variability
was such that the statistical confidence level was judged
to be poor. It has been concluded that by measuring ultra-
violet reflectance the spectrophotometer can indeed be used
as a non-destructive tool to detect whether or not a stored
nylon parachute has reached a dangerous and unsafe level of
degradation. However, if it is necessary to determine exact
extents of damage, tensile tests still appear to be necessary.

ASD TR 61-667

November 1961

SUBJECT: FLEXIBLE LOW POROSITY, WOVEN METALLIC
MATERIALS

INVESTIGATOR: M. J. Coplan, W. D. Freeston, Jr., D.
H. Powers, Jr.

CONTRACT: AF33(616)-7222, Fabric Research Labs, Inc.

ABSTRACT: The design of numerous re-entry drag or
lift-drag devices incorporates a flexible, low porosity,
thermally durable membrane. The missions anticipate tempera-
tures in the range of 1500 to 2500°F and strengths of 20 to
50 lbs/inch width. The necessity of being able to package
and subsequently deploy these devices requires a membrane
with good bending recovery also. These design requirements
indicate a fabric woven from stranded yarns of fine super
alloy or refractory metal filaments as the most promising
solution at the present time. Monofilament screen fabrics
have too low a flexibility and too high a porosity to be
suitable.

ASD TR 61-677 (Continued)

The feasibility of stranding and plying into yarns metal filaments as fine as 1/2 mil and subsequently weaving the yarns has been shown. Flexible, thermally durable fabrics with extremely low porosities have been obtained.

A possible application of the metamorphic principle has been demonstrated by the high temperature exposure testing of a fabric woven from a composite yarn composed of a chemically treated organic yarn and 1/2 mil metal filaments.

Flexible, nonporous coatings have been applied to high temperature fabrics. These coatings consist of a flake material, mica, bonded to the fabric with silicone varnish.

ASD TR 62-180

February 1962

SUBJECT: METAL FILAMENTS FOR HIGH-TEMPERATURE FABRICS
INVESTIGATOR: D. E. Johnson, E. H. Newton, E. R. Benton
CONTRACT: AF33(616)-7294, Arthur D. Little, Inc.
ABSTRACT: This research centered primarily on the oxidation and tensile properties of 0.5- to 5.0-mil filaments of three superalloys (Elgiloy, Rene 41 and Inconel 702) and two refractory metals (tungsten and molybdenum) in the 1500-2000°F temperature range. The oxidation data for the refractory metals are for ultra-short-time durations, up to 100 milliseconds. Preliminary creep rupture data are also given for the above three superalloys, along with some data for Karma and Nichrome V.

Attempts to develop very thin (0.05- to 0.1-mil) oxidation-resistant coatings for molybdenum and tungsten by the use of various plating techniques are also reported.

One-half-mil fibers of superalloy wire were woven into fabrics of various constructions; the influence of fabric construction on permeability and aerodynamic drag (at Mach 4.8) is discussed.

The literature pertaining to heat transfer to single cylinders in hypersonic flow is reviewed, and

ASD TR 62-180 (Continued)

sample calculations of equilibrium temperature are made to point up areas where the data are deficient.

This report also presents the results of preliminary research on three new techniques for forming fine filaments without the use of a diamond die.

ASD TDR 62-185

February 1962

SUBJECT: THERMAL RADIATION EFFECTS ON DECELERATOR MATERIALS
INVESTIGATOR: P. E. Glaser, S. Merra
CONTRACT: AF33(616)-7692, Arthur D. Little, Inc.
ABSTRACT: An investigation was made of the effects of thermal radiation on the strength of fibrous materials used in deceleration devices.

Approximately 400 tests were run on webbing and tape materials of various strengths. The work involved the development of laboratory equipment to simulate the thermal shock of a nuclear blast, flux redistributors, a high-velocity air supply, and a static loading device.

It was found that the strength of a given material is rapidly degraded at heat fluxes above a critical value. The critical flux, which varies with the material, is substantially reduced by slight soiling. Heat resistance is not affected by placing the sample under tension but is improved by air cooling. It is also strongly influenced by the type of weave.

FUELS, LUBRICANTS, AND FLUIDS

WADD TR 60-898

March 1961

SUBJECT: FLUIDS, LUBRICANTS, FUELS, AND RELATED MATERIALS

INVESTIGATOR: E. Erwin Klaus, M. R. Fenske, E. J. Tewksbury

CONTRACT: AF33(616)-5460, Pennsylvania State Univ.

ABSTRACT: This report describes work carried out on a continuing program to characterize the capabilities of hydraulic fluids, lubricants, and functional fluids for aeronautic and astronautic applications under extreme environmental conditions. The effects of solvent type and solvent to oil ratio on the deep de-waxing process are shown. The yield and viscosity-temperature properties of the deep de-waxed oil are related to the type and degree of refining of the mineral oil fraction. The preparation of large volumes of super-refined mineral oil formulations for "mock-up" testing is reported. Extensive technical liaison on processing, properties, and application is discussed. Physical and chemical stability of base stocks, additives and finished hydraulic fluid and lubricant formulations after 5 to 17 years in storage is described. A sample of hydraulic fluid taken from the "Lady Be Good" B-24 Bomber after 16 years in the North African desert is discussed in detail. The design, construction, and preliminary testing of a versatile capillary pressure viscometer is reported. The use of this viscometer to measure the effect of gas solubility on viscosity and the analysis of flow profile in a capillary viscometer is discussed. The use of this pressure unit with a modified Lipkin pycnometer for the measure of bulk modulus is suggested. The thermal stability of esters are contrasted and compared as a function of chemical structure. Quantitative evaluations of the gas produced and the liquid phase are used to illustrate the effect of metal catalysts. The effects of fluid type, viscosity, vapor pressure, oxidation mechanism, oxidation inhibitor, and gaseous environment on evaporation are presented. The use of evaporation tests in studying the mechanism of oxidation is suggested. The relative lubricity properties of a series of high temperature bearing materials are reported. The relative effects of fluid volatility on lubricity are discussed. The similarities between high temperature lubricity properties and the low temperature lubricity properties of the residual fluids after high temperature oxidation and thermal tests are pointed out. The wear properties of mineral oils and esters with and without lubricity additives are compared and con-

WADD TR 60-898 (Continued)

trasted with silicone and silicate fluids over the temperature range of 167° to 700°F. A simple versatile, quantitative oxidation test is described for use with a variety of high temperature oxidation tests.

FUELS

WADD TN 60-238, OTS Release

December 1960

SUBJECT: THE EFFECT OF PARA-ORTHO SHIFT ON HEAT EXCHANGER DESIGN IN HYDROGEN NITROGEN HEAT EXCHANGE AT HIGH PRESSURES

INVESTIGATOR: J. H.L. Lawler

ABSTRACT: The effect of para-ortho shift is found on heat exchanger design by calculating heat exchanger sizes for several hydrogen to nitrogen flow ratios. This calculation shows that size is exponential to a limit when plotted against lbs of H₂/lbs of N₂(#H₂) flow and that para-ortho shift has a pronounced effect above 5#H₂/#N₂.

WADD TN 60-254, OTS Release

November 1960

SUBJECT: THE SPECIFIC IMPULSE OF HYDROGEN BURNED IN NITROGEN-OXYGEN MIXTURES

INVESTIGATOR: J. H. L. Lawler

ABSTRACT: Specific impulse curves are presented for oxygen-nitrogen mixtures of 90, 85, 80, 75, 70, and 60% by weight oxygen being burned with hydrogen. The chamber pressures were 1000 psia and 500 psia and various exhaust pressures from 7.35 psia to 0.005 psia were used. Expansion ratio vs. impulses for the several conditions is plotted.

WADD TR 60-766, OTS Release

November 1960

SUBJECT: STUDY OF PHYSIOCHEMICAL PROPERTIES OF COMMERCIAL SAMPLES OF ISOPROPYLBICYCLOHEXYL AND DIETHYLCYCLOHEXANE

INVESTIGATOR: E. Findl, H. Brande, H. Edwards

CONTRACT: AF33(616)-5587, Thompson Products, Inc.

ABSTRACT: A study was made of certain physical and thermodynamic properties of commercial samples of isopropylbicyclohexane and diethylcyclohexane. Isopropylbicyclohexane is quite similar to fuels meeting with RJ-1 specification in most of its thermodynamic properties.

WADC TR 53-373 Sup 9

131

SUBJECT: STUDY OF PHYSIOCHEMICAL PROPERTIES OF
SELECTED MILITARY FUELS

INVESTIGATOR: E. Findl, H. Brande, H. Edwards

CONTRACT: AF33(616)-3729, Thompson Products, Inc.

ABSTRACT: The purpose of the program was to determine certain physical and thermodynamic properties of four selected aircraft fuels. These fuels were MIL-F-5624C Grade JP-4, a MIL-F-25558 Grade RJ-1*, MIL-F-25656 Grade JP-6-H, and Decalin, a commercial hydrocarbon mixture of the cis and trans isomers of decahydronaphthalene. Specific properties evaluated were: 1. Equilibrium solubility of air, nitrogen and ethane in the JP-4 and RJ-1 Fuels. 2. Effect of dissolved air, nitrogen, and ethane on the viscosity of the JP-4 and RJ-1 Fuels. 3. Effect of dissolved ethane on density of the JP-4 and RJ-1 Fuels. 4. Effect of pressure level, agitation rate, and rate of pressure change on the evolution rate of air and ethane from the JP-4 and RJ-1 Fuels. 5. Thermodynamic properties of each of the four test fuels including specific heats, enthalpy, and entropy. 6. Vaporization characteristics of each of the test fuels including vapor pressure, equilibrium vaporization curves, minimum reflux curves, variation of vapor molecular weight during minimum reflux distillations. 7. Variation of liquid density as a function of temperature. 8. Literature survey correlation compilation for the determination of physical and thermodynamic properties of petroleum fuels.

*Formerly designated as Shell UMF, Grade C.

SUBJECT: EVALUATION OF MATERIALS AS ENDOTHERMIC
AVIATION FUELS

INVESTIGATOR: B. M. Fabuss, A. S. Borsanyi, J. L.
Driscoll, B. J. Gudzinowicz, R. I. Lait,
J. O. Smith, C. F. Wurster, Jr.

CONTRACT: AF33(616)-6608, Monsanto Chemical Co.

ABSTRACT: In order to determine the feasibility of using endothermic hydrocarbons (hydrocarbons that not only absorb sensible heat while being heated but also undergo heat-absorbing changes in structure of composition) as heat sink aviation fuels and to increase their heat absorption capacity, the endothermic thermal cracking of cetane was investigated. A small-scale laboratory cracking unit was assembled and operated to 1400°F at pressures up to 1000 psig, and with liquid fuel feed rates up to 8 liters per hour. An experimental program involving cracking at all combinations of three different temperatures, pressures, and flow rates was completed.

December 1961

SUBJECT: EVALUATION OF MATERIALS AS ENDOTHERMIC AVIATION FUELS

INVESTIGATOR: J. O. Smith, B. M. Fabuss, A. S. Borsanyi, R. I. Laid

CONTRACT: AF33(616)-7845, Monsanto Research Corp.

ABSTRACT: Pure cetane, cyclohexane, ethylbenzene, p-xylene, decalin, Soltrol-170, and tetraisobutylene were evaluated as potential endothermic fuels. Cetane, decalin, Soltrol-170, and cyclohexane can be used as endothermic fuels from 1100 to 1300°F. Tetraisobutylene is best from 800 to 900°F. The heat sink capacity of cetane, cyclohexane, decalin, and Soltrol-170 is 1130 to 1250 Btu/lb at 1300°F wall temperature and 60% conversion; that of tetraisobutylene is 880 to 950 Btu from 800 to 900°F. Direct measurement of heat inputs showed that the calculated heats of reaction are of the proper order. The feasibility of coke removal from the reactor tube walls by steam injection was established. Three paraffinic CRC fuels were evaluated. Their heats of reaction are 100-400 Btu/lb of converted material at 40-60% conversion; the heat sink capacity is 1050 to 1250 Btu/lb at 1300°F wall temperature and 60% conversion.

WADD TR 61-89

May 1961

SUBJECT: FLAMMABILITY CHARACTERISTICS OF HIGH TEMPERATURE HYDROCARBON FUELS

INVESTIGATOR: J. M. Kuchta, A. Bartkowiak, I. Spolan, M. G. Zabetakis

CONTRACT: D033(616)-60-5, United States Department of the Interior

ABSTRACT: Data are presented on the flammability and auto-ignition temperature characteristics of JP-6, JP-150 and HTF-59-24 fuels. Flammability limits of the vapors of these fuels were determined in air at elevated temperatures and at atmospheric pressure. Auto-ignition temperatures and corresponding ignition delay times were obtained under static and dynamic conditions as a function of the pressure and the oxygen concentration of the ambient medium; the effect of fuel volume on auto-ignition was also determined in some static tests. In addition, the auto-ignition of JP-6 fuel vapor-oxygen-nitrogen mixtures was studied in various vessels which were heated uniformly and non-uniformly; critical heat flow requirements for auto-ignition were found for spherical and cylindrical copper vessels and a spherical stainless steel vessel.

ASD TR 61-193

June 1961

SUBJECT: DEVELOPMENT OF MICROBIOLOGICAL SLUDGE INHIBITORS
INVESTIGATOR: A. V. Churchill, W. W. Leathen
CONTRACT: AF33(616)-6989, Gulf Research & Development Company
ABSTRACT: A research investigation was carried out to develop information and materials for control of microbiological sludge formation in jet fuel bulk storage tanks. A total of 184 microorganisms were isolated from jet fuel-water samples; 61% of the isolates survived viability tests. They appeared to fall into eight distinct groups consisting of five fungi (a brown type predominated) and three bacteria (transparent and opaque types predominated). Approximately 178 water-soluble materials were evaluated as potential microbiological sludge inhibitors. Three compounds are recommended for trial in bulk storage tanks to control microbial growth. These are alkyl quaternary ammonium acetate, ethylidene diacetate and tri-n-butyl borate. Several others satisfactorily controlled microbial growth but contained elements that are potentially deleterious to fuel properties and fuel system materials. No appreciable changes in properties of JP-4 jet fuel were noted in storage tests of 90 days' duration involving jet fuel-mineral salts substrates with added microorganisms.

ASD TR 61-266

November 1961

SUBJECT: RESEARCH AND DEVELOPMENT ON THERMAL STABILITY OF SPECIAL HYDROCARBON FUELS
INVESTIGATOR: R. Wolfshagen, H. Braude, W. Welge
CONTRACT: AF33(616)-7275, Thompson Ramo-Wooldridge
ABSTRACT: The thermal stabilities of various fuels for use in Mach 3 to Mach 4 aircraft were evaluated in an aircraft fuel system-engine thermal simulator. Test conditions simulated heat flux and fuel temperature levels anticipated in high Mach aircraft. Included were simulation of heat input from aerodynamic fuel tank heating, hydraulic and lube oil heat exchangers and engine nozzle environment sources. Five fuels, all JP-6 types, were tested under simulated Mach 3 conditions, and five higher temperature fuels, including several nearly pure hydrocarbons, were tested under simulated Mach 4 conditions. The fuels were rated on the basis of relative change of heat exchanger performance in 50 operating hours. The fuels were also rated in the ASTM-CFR Fuel Coker (HP-6 type fuels) and the Erdco Research Coker (higher temperature fuels) to obtain an evaluation of this test method with respect to simulator results.

WADC TR 53-373 Sup 9

134

ASD TR 61-360

October 1961

SUBJECT: THERMODYNAMIC PROPERTIES OF 20.4°K-EQUILIBRIUM HYDROGEN
INVESTIGATOR: A. Shaffer, J. Rousseau
CONTRACT: AF33(616)-7895, AiResearch Mfg. Co.
ABSTRACT: This report presents the thermodynamic properties of 20.4°K-equilibrium hydrogen for pressures between 1 psia and 3000 psia and temperatures between 25°R and 5000°R. Several graphical representations of the data are given. These consist of temperature-entropy, enthalpy-entropy, pressure-enthalpy, temperature-enthalpy, and temperature-density diagrams. Data are given to show the effect of ortho-para composition on hydrogen properties over the range of temperatures considered. High-temperature gas properties shown include the effects of dissociation and non-ideal gas behavior.

ASD TR 61-536

September 1961

SUBJECT: SATURATION PROPERTIES OF OXYGEN NITROGEN MIXTURES
INVESTIGATOR: E. F. Yendall, W. J. Olszewski
CONTRACT: AF33(616)-8291, Linde Co.
ABSTRACT: A correlation for the relative volatility of the oxygen nitrogen system was developed using data available in the literature and previously unpublished Linde Company data. The heat contents of the saturated vapors and liquids were computed using an equation-of-state approach for the vapor heat content, and the latent heats as obtained from the Clausius-Clapeyron equation, to determine the liquid heat contents.

The results have been tabulated and plotted for the pure gases and the mixtures at 10% increments as large scale graphs of relative volatility as a function of temperature and pressure, temperature-composition at fixed pressures, and a Mollier diagram of pressure versus enthalpy. All the charts extend from atmospheric to critical pressures.

HYDRAULIC FLUIDS

WADD TR 60-838, Part I

March 1961

SUBJECT: THE SYNTHESIS AND EVALUATION OF NEW BASE STOCK FLUIDS FOR GAS TURBINE APPLICATION

INVESTIGATOR: J. D. Behun

CONTRACT: AF33(616)-6749, Wyandotte Chemicals Corp.

ABSTRACT: The synthesis and evaluation of pyrazine compounds are described. Using 2-chloro-3-methylpyrazine, successful nucleophilic displacement reactions were performed to replace the chlorine with different phenoxide, alkyl- and arylmercapto, arylamino and silyl substituents. The resultant 2-substituted 3-methylpyrazine intermediates were then further modified at the methyl site. Many of the 2,3-disubstituted pyrazine model compounds are liquids at room temperature.

WADD TR 60-838, Part II

February 1962

SUBJECT: THE SYNTHESIS AND EVALUATION OF NEW BASE STOCK FLUIDS FOR GAS TURBINE APPLICATION

INVESTIGATOR: P.T. Kan

CONTRACT: AF33(616)-6749, Wyandotte Chemicals Corp.

ABSTRACT: The synthesis and evaluation of new pyrazine derivatives for high temperature stable fluid applications is given. Candidate products, unsymmetrical 2,3-disubstituted- and 2,3,6-trisubstitutedpyrazines were prepared from 2-chloro-3-methylpyrazine and 2-chloro-3,6-dimethylpyrazine. Alkyl substituted pyrazines were synthesized from 2,5- and 2,6-dimethylpyrazines and tetramethylpyrazine. A wide variety of pyrazine derivatives were obtained in good to excellent yields.

These compounds were screened for thermal and oxidative stability. Classes of compounds containing undesirable linkages were bypassed and materials which showed promise were screened further. Correlations of the effect of variation in structure upon physical properties of the substituted pyrazines were made. Using these correlations, a stepwise achievement of the candidate products with further improved properties was realized.

WADD TR 60-901, OTS Release

January 1961

SUBJECT: ION PROPULSION WORKING FLUID REQUIREMENTS
FOR USAF
INVESTIGATOR: J. H. L. Lawler
ABSTRACT: Some of the anticipated requirements for
ion engine working fluids are derived by the consideration
of missions expected to be of interest, the probable systems
which will produce the power, and handling needs.

ASD TR 61-405, Part I

December 1961

SUBJECT: DEVELOPMENT OF FIRE RESISTANT HYDRAULIC
FLUIDS
INVESTIGATOR: C. Popoff, R. K. Smith
CONTRACT: AF33(616)-7457, E. F. Houghton & Co.
ABSTRACT: The development of fluoroalkyl substituted
silicates has been explored. Synthesis of prototype materials
was directed to the study of reaction systems through trans-
esterification of fluoroalkyl alcohols with tetraethyl sili-
cate. The reactions of silicon tetrachloride and hexachloro-
disiloxane with fluoroalkyl alcohols was also studied.

The most promising fluid was derived
from the reaction of perfluoroheptanol with hexachlorodisi-
loxane. The product was stable at 650°F, had a pour point
of -40°F and had good resistance to oxidation. The dis-
advantages of the fluid were poor hydrolytic stability and
high density.

ASD TDR 62-259, OTS Release

April 1962

SUBJECT: $\bar{J} \times \bar{B}$ MAGNETOHYDRODYNAMIC (MHD) WORKING
FLUIDS
INVESTIGATOR: M. C. Gourdine
CONTRACT: AF33(616)-8173, Plasmadyne Corp.
ABSTRACT: A literature survey covering the three
types of MHD propulsors (shunt, series, induction) is pro-
vided. A theory suitable for analyzing the performance of
the steady $J \times B$ accelerator is developed. The performance
of various working fluids is determined using the criterion
of maximum propulsion efficiency. Two broad classes of
working fluids are considered: equilibrium plasmas, and non-
equilibrium plasmas. Non-equilibrium plasmas have the ad-
vantage of good performance without high temperature materials
problems. A conductivity of 170 mhos/m in argon has been ex-
perimentally produced and measured in a "ring" discharge at

ASD TDR 62-259 (Continued)

a pressure of 1 mm Hg with a power input approximately 1 kilowatt. A temperature of over 5,000°K is required to do this thermally.

HEAT TRANSFER FLUIDS

WADD TR 60-795, Part II

February 1962

SUBJECT: EXTENDED STUDY OF -80°F TO 400°F
TEMPERATURE RANGE FLUIDS

INVESTIGATOR: D. A. Barsness

ABSTRACT: The information contained in this report is based on research leading to new and improved extreme temperature range (ETR) coolants for electronic equipment. This is a continuation study of the thermal and electrical properties of the four candidate -80°F to 400°F materials as discussed in WADD TR 60-795, Part I. Research was also continued on new experimental materials of an additional silicone fluid which possesses desirable properties for use over the aforementioned temperature range. These fluids are intended to operate as single liquid phase coolants at nominal ambient pressures throughout the specified temperature range and to have desirable electrical and chemical properties. They may be used as primary heat transfer fluids in an electronic or aerospace system.

WADD TR 61-96

November 1961

SUBJECT: PROPERTIES OF INORGANIC ENERGY-CONVERSION
AND HEAT-TRANSFER FLUIDS FOR SPACE
APPLICATIONS

INVESTIGATOR: W. D. Weatherford, Jr., J. C. Tyler,
P. M. Ku

CONTRACT: AF33(616)-7206, Southwest Research Inst.

ABSTRACT: This report is a complete revision of WADC TR 59-598. It is intended to serve as a properties handbook for various inorganic fluids which may have potential value as energy-conversion or heat-transfer fluids for space applications. The fluids are presented as three distinct classes - namely, liquid metals, non-metals, and gases. The liquid metals include mercury, cesium, rubidium, potassium, NaK (78), sodium, lithium, bismuth, and lead. The non-metals include aluminum bromide, sulfur, and lithium hydride. The gases include argon, helium, and hydrogen.

WADC TR 53-373 Sup 9

138

WADD TR 61-96 (Continued)

Data are presented, where available, up to temperatures ranging from 2300°F for mercury to 4500°F for lead, and for pressures ranging from less than one atmosphere to greater than twenty atmospheres. The enumerated properties include vapor pressure, density, viscosity, surface tension, electrical resistivity, thermal conductivity, specific heat, latent heats, enthalpy-entropy relationships, melting point, critical properties, dielectric constant, ionization potential, magnetic susceptibility, thermal neutron cross sections, and corrosion characteristics.

WADD TR 61-186

July 1961

SUBJECT: ADVANCED HEAT TRANSFER FLUIDS
INVESTIGATOR: L. J. Martin, C. W. Mell, J. T. Milek
CONTRACT: AF 33(616)-7109, Hughes Aircraft Co.
ABSTRACT: An electronic coolant heat transfer study of organic fluids from -80°F to 400°F. Silicone fluids appear to be the most promising over the temperature range of -80°F to 400°F. A dynamic loop apparatus was devised and built to permit an indication of the thermal chemical-dielectric-time stability of these coolants in electronic devices and equipment used in ground-based, ship-based and satellite vehicles. An extensive literature survey on electronic coolants, electronic equipment cooling, heat transfer, dielectric properties and stability, thermal stability, test methods and apparatus, liquid metals and fused salts for liquid phase and liquid-vapor cycle cooling.

WADD TR 61-259

May 1961

SUBJECT: MATERIALS RESEARCH FOR LUBRICANTS AND HEAT TRANSFER FLUIDS
INVESTIGATOR: K. R. Mecklenburg
CONTRACT: AF33(616)-7336, Midwest Research Inst.
ABSTRACT: A discussion of the design concepts of an apparatus to boil and condense liquid metals, liquid metal salts, non-metals is presented along with schematics and drawings. Viscosity, density-temperature, surface tension, and flash, fire, and pour points data for extreme temperature range for electronic gear coolants are presented and discussed.

Heat requirements for a high speed bearing apparatus were determined and the necessary modifications

WADD TR 61-259 (Continued)

were made to extend the operating temperature to 700°F. Greases were studied to determine their life at temperatures from 350 to 450°F. Experimental agreement of the IAE gear lubricant testing machine data is discussed. The design of a vacuum dry film apparatus is discussed along with certain operating features. Results of lubricant screening studies using the Four-Ball Wear Tester are presented, and the modifications made to extend the operating range of the Four-Ball Wear Tester are discussed.

LUBRICANTS

ASD TR 54-576, Part V

April 1961

SUBJECT: EFFECT OF METALS ON THE THERMAL AND OXIDATIVE STABILITY OF LUBRICANTS AT ELEVATED TEMPERATURES

INVESTIGATOR: D. C. Trop

ABSTRACT: Silicone, silane, and mineral oil lubricating fluids were studied at 700°F under inert and oxidizing atmospheres while in the presence of various individual metals. Physical and chemical changes in the fluids and metals were noted. Fluids studied were two (2) methyl chlorophenyl silicones, a methyl phenyl silicone, a hydrogenated mineral oil and a silane. Metals used included magnesium, titanium, aluminum, silver, copper brass, bronze, monel steel, stainless steel, copper-beryllium steel, vanadium tool steel, chrome plated steel, cast iron and chrome-molybdenum steel. The silane fluid was the least affected fluid and was also the most compatible fluid with metals.

WADC TR 58-288, Part IV

August 1961

SUBJECT: DEVELOPMENT OF HIGH TEMPERATURE HEAVY LOAD CARRYING GREASES

INVESTIGATOR: R. K. Smith, S. Braid, C. Popoff

CONTRACT: AF33(616)-7320, E. F. Houghton & Co.

ABSTRACT: The object of this work was to continue the development of high temperature extreme pressure grease systems. The study of new base fluids and extreme pressure additive has been performed utilizing previously developed conjugated metallo organic thickening agents.

WADC TR 53-373 Sup 9

140

WADC TR 58-350, Part IV

September 1961

SUBJECT: DEVELOPMENT OF GREASES FOR HIGH SPEED BALL AND ROLLER BEARINGS
INVESTIGATOR: P. R. McCarthy, J. F. Hedenburg, E. R. Dzuna
CONTRACT: AF33(616)-6963, Gulf Research & Dev. Co.
ABSTRACT: The objective of this work was the development of a grease capable of lubricating anti-friction bearings at 600°F and at speeds between 20,000 and 45,000 rpm. The initial target requirement was 100 hours of operation at 20,000 rpm.

A wide variety of materials were evaluated as potential fluids and thickeners. An experimental grease containing ammeline as the thickener and a silicone as the fluid met the initial life requirement. At 45,000 rpm life was short, ranging from 0 to 9 hours. Neither the type of grease, fluid used in the grease nor type of bearings had an appreciable effect on life at this speed.

WADC TR 59-603, Part II

May 1961

SUBJECT: FRICTION AND WEAR AT ELEVATED TEMPERATURES
INVESTIGATOR: E. Rabinowicz, M. Imai
CONTRACT: AF33(616)-5963, Mass. Inst. of Tech.
ABSTRACT: Friction experiments in the range room temperature to 1800°F have been carried out on a wide variety of materials. Many of the results fall into no particular pattern, but tests involving boron carbide show a sharp peak in the friction-temperature plot. This is traced to the influence of boric oxide formed by oxidation. Studies of the properties of boric oxide suggest that it may be a useful boundary lubricant at elevated temperatures.

WADD TR 60-529, Part II

June 1961

SUBJECT: LUBRICATION STUDIES WITH GRAPHITE SINGLE CRYSTALS
INVESTIGATOR: P. J. Bryant
CONTRACT: AF33(616)-6277, Midwest Research Inst.
ABSTRACT: A basic research program to determine the mechanisms of lubrication and wear for the fundamental graphite system is being conducted with single crystalline graphite samples. Graphite single crystals were grown by two methods and compared to natural crystals. The atomic spacings of the various graphite samples were examined by

WADD TR 60-529, Part II (Continued)

X-ray diffraction; the dislocation structure, the impurity and vacancy content were studied by means of electron microscopy. The stacking fault energy of the grown crystals is higher than that for natural graphite indicating a significant difference in van der Waals' forces. An ultra-low pressure controlled atmosphere test chamber has been designed and constructed for measurements of the energy of cohesion of graphite versus atmospheric content. The chamber has produced unusually low pressure (10^{-12} mm. Hg) and good sample manipulation.

WADD TR 60-728, Part I

March 1961

SUBJECT: LUBRICANT FOR HIGH-VACUUM ENVIRONMENT
INVESTIGATOR: M. M. Freundlich, S. S. Jagodowski
CONTRACT: AF33(616)-6845, Airborne Instruments Lab
ABSTRACT: The purpose of the work was to determine the possibility of using available oil lubricants in bearings of small motors that have to operate for many months inside space vehicles. The rate of evaporation of lubricants impregnated into rotating and stationary ball bearings was measured in high vacuum. A high-vacuum microbalance was built which permitted measurement of the rate of evaporation of different lubricating oils at various temperature levels during the evaporation of the sample, without the need to break the vacuum. It was found that (1) initial rate of evaporation was practically independent of the speed of rotation, (2) the rate of evaporation was proportional to the bearing size, (3) the rate of evaporation of shielded bearings was only about 15 percent lower than that of unshielded bearings, (4) the evaporation rate decreased by many orders of magnitude from the start of the evaporation until the end when the material had evaporated completely. It is recommended to measure the evaporation rates of other oil lubricants and to test the lower fractions only of the best oils in motors operating in high vacuum.

WADD TR 60-728, Part II

February 1962

SUBJECT: LUBRICANT FOR HIGH-VACUUM ENVIRONMENT
INVESTIGATOR: M. M. Freundlich, S. J. Jagodowski
CONTRACT: AF33(616)-6845, Airborne Instruments Lab
ABSTRACT: The objective of this work was to determine vapor pressures and evaporation rates of four experimental high-temperature fluids in a high-vacuum en-

WADD TR 60-728, Part II (Continued)

vironment over a wide range of temperatures. The vacuum microscale developed in Part I of this investigation was improved and the measurement method refined. The microscale was checked with n-heptadecane and the results showed good agreement with published values. The following four fluids were supplied by ASD and their evaporation-rate and vapor-pressure curves are given: (1) hexaphenyl ether, (2) Siloxane, (3) silicone fluid QF-6-7040 and (4) silicone fluid F-6-7024.

WADC TR 60-753, Part I

March 1961

SUBJECT: NUCLEAR RADIATION RESISTANT GYROSCOPE
BEARING LUBRICANTS AND FLOTATION MEDIA
INVESTIGATOR: R. A. Falk
CONTRACT: AF33(616)-6817, Sperry Gyroscope Co.
ABSTRACT: Available data concerning various classes of organic liquids are presented and discussed with respect to radiation resistance and potential application as gyro lubricants and flotation fluids. A number of these fluids were selected as lubricant candidates and additional evaluation performed. Suitable radiation resistant flotation fluids are not currently available; therefore, a synthesis program was initiated to prepare such materials. The study and evaluation of existing lubricants and synthesis of flotation fluids are reported herein.

WADD TR 60-753, Part II

February 1962

SUBJECT: NUCLEAR RADIATION RESISTANT GYROSCOPE
BEARING LUBRICANTS AND FLOTATION MEDIA
INVESTIGATOR: F. R. Callihan
CONTRACT: AF33(616)-6817, Sperry Gyroscope Co.
ABSTRACT: Fluids for use as base stocks for the formulation of radiation-resistant lubricants were investigated; one of them tert-butyl 1, 9-diphenylnonane, appears particularly promising and is recommended for further evaluation.

Towards the objective of developing perfluoroaromatic materials as highly stable fluids, methods for the preparation of hexafluorobenzene were investigated. A successful procedure involving the pyrolysis of dichlorofluoromethane was developed and is reported herein.

WADD TR 60-855, Part II

March 1962

SUBJECT: LUBRICATION BEHAVIOR AND CHEMICAL DEGRADATION CHARACTERISTICS OF EXPERIMENTAL HIGH TEMPERATURE FLUIDS AND LUBRICANTS

INVESTIGATOR: V. Hopkins, A. D. St John, D. Wilson

CONTRACT: AF33(616)-6854, Midwest Research Inst.

ABSTRACT: MLO 60-294 resisted degradation from high shear stresses at 400°, 500°, 550°, and 600°F and wear of the hydraulic pump was small through 550°F. MLO 59-91 at 400°F permitted rapid wear in the hydraulic pump. MLO 59-692 was not degraded by high shear stresses at 550° and 700°F. QF-258 was not degraded at 550°F but experienced a drop in viscosity and flash point during a 100 hr. shear stability experiment at 700°F. Bulk modulus data are presented for MLO 60-294 and QF-258. Results of lubricant behavior in a rolling-sliding contact are presented and a partial analysis of roller-cage stability is given. Development of the high pressure viscometer is discussed. Solid film lubrication of spherical bushings and the effects of thermal aging of a film is presented. Extreme pressure lubrication of M-10 tool steel at 400°F and 600°F and of 52100 steel at 275° and 400°F with an ester of TMP with various additives is discussed.

WADD TR 60-898, Part II

February 1962

SUBJECT: FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS

INVESTIGATOR: E. E. Klaus, M. R. Fenske, E. J. Tewksbury

CONTRACT: AF33(616)-7590, Pennsylvania State Univ.

ABSTRACT: Report describes continuing program to characterize capabilities of fluids and lubricants for aeronautic or astronautic uses. Feasibility of commercial production of low temperature mineral oils is indicated. Formulations dependent on end use are suggested. Excellent blending efficiency for mineral oil-ester blends are shown. Data for phenyl ethers are included. Data suggest use of radioisotopes as an aid in measurement of relative polarity of additives, in establishment of lubrication mechanisms, and in analysis for impurities. Equipment and procedures for measurement of bulk modulus are described. A series of pressure-viscosity determinations is shown. Behavior of fluids in a system having a large pressure or temperature differential across a small leak or vent is discussed. Design and construction of units for measurement of heat

WADC TR 53-373 Sup 9

144

WADD TR 60-898, Part II (Continued)

capacity and thermal conductivity are illustrated. Prediction of useful life for fluids based on oxidation behavior is illustrated. Special M-2 tool steel bearings are evaluated. Stability of aromatic hydrocarbons is discussed.

WADD TR 60-913

March 1961

SUBJECT: THE SYNTHESIS AND EVALUATION OF AROMATIC ESTERS AS POTENTIAL BASE STOCK FLUIDS FOR GAS TURBINE ENGINE LUBRICANTS

INVESTIGATOR: W. E. Taylor, E. R. Witt, C. L. Osborn

CONTRACT: AF33(616)-6786, Celanese Chemical Co.

ABSTRACT: A number of carboxylic acid esters were synthesized and evaluated as base stock fluids for lubricants suitable for operation at bulk oil temperatures of 450°F to 500°F. Oxidation stability at these temperatures is required and suitable fluidity at -65°F is desired. Esters were prepared from aromatic and aliphatic acids with phenols, benzyl type alcohols and 2,2-dimethylalkyl aliphatic alcohols. Their physical properties and thermal stabilities were determined and could be correlated with their structural units. Phenol esters of aromatic and 2,2-dimethylalkyl carboxylic acids were thermally stable at 750°F and oxidatively stable at 450°F without inhibitors; esters of non-alkylated phenols gave the best oxidative stabilities. Low temperature properties were very poor for the aromatic acid esters; their pour points were in excess of 70°F and many were high melting solids. Esters of 2,2-dimethylalkyl acids gave fluids with pour points well below this level. One of these fluids, resorcinol dineoheptanoate, showed, when inhibited, excellent resistance to oxidation at 475°F. Its volatility was somewhat high and its pour point (-55°F) above the target property, but it demonstrated the possibilities of obtaining an ester fluid with the range of desired properties. Suggestions are given for future studies aimed at developing a similar but less volatile ester.

WADD TR 60-913, Part II

January 1962

SUBJECT: THE SYNTHESIS AND EVALUATION OF AROMATIC ESTERS AS POTENTIAL BASE STOCK FLUIDS FOR GAS TURBINE ENGINE LUBRICANTS

INVESTIGATOR: W. E. Taylor, C. L. Osborn, N. F. Swynnerton

CONTRACT: AF33(616)-6786, Celanese Chemical Co.

ABSTRACT: The synthesis and evaluation are reported

WADC TR 53-373 Sup 9

145

WADD TR 60-913, Part II (Continued)

for several new carboxylic acid esters, prepared as lubricant base stock candidates for operation at bulk oil temperatures of 450-500°F. The esters were prepared from various combinations of 2,2-dimethylalkyl acids, n-alkyl and aromatic dibasic acids plus alkylated phenoxyphenols, dihydroxydiphenyl ethers, resorcinol, alkylphenols and neopentyl glycol (2,2-dimethyl-propane-1,3diol). The physical properties and oxidative and thermal stabilities of these esters were determined and are correlated with their structural configuration. Several of the esters were thermally stable at 750°F and resistant to autoxidation at 450°F. Esters of 2,2-dimethylpentanoic acid and substituted phenoxyphenols had pour points ranging from -3° to + 28°F. One neopentyl glycol ester, di-2,2-dimethylpropane-1,3-diol mono-2,2-dimethylpentanoate) azelate, was thermally stable above 700°F and, with inhibitors, resistant to oxidation at 450°F. This ester has a pour point of -40°F. Suggestions are made for future work with esters of similar structure to increase their stability.

WADD TR 60-916

August 1961

SUBJECT: HEAT CAPACITY AND THERMAL CONDUCTIVITY
OF LUBRICATING OILS AND HYDRAULIC FLUIDS
INVESTIGATOR: D. C. Trop
ABSTRACT: The heat capacities of 36 lubricating oils and hydraulic fluids (polyphenyl ethers, disiloxanes, silanes, TMP esters, and silphenylene) were measured over a temperature range of 80° to 500°F by adiabatic and mixture methods. The "hot wire" and concentric cylinder types of thermal conductivity cells were fabricated and calibrated to measure the thermal conductivity of 18 lubricating oils and hydraulic fluids. Both methods are suitable for accurate thermal conductivity determinations.

WADD TR 61-8

April 1961

SUBJECT: HIGH TEMPERATURE GEAR AND SPLINE LUBRICANT STUDIES
INVESTIGATOR: D. J. Fessett
CONTRACT: AF33(616)-5631, Western Gear Corp.
ABSTRACT: Studies were conducted on lubricants applied to spur gears at speeds to 20,000 rpm, lubricant temperatures to 800°F and pressure simulating altitudes to 80,000 ft. Lubricants tested in order of their decreasing

WADD TR 61-8 (Continued)

load carrying ability (score load) at 400°F are: MLO 7452 (mineral oil), Octadecyl tri(decyl) silane, MLO 58-431 (ester silicone), 57-426 (ester), 7379, 7381, 7380, 7383 (mineral oils), and 59-692. Endurance studies conducted on the lubricants at the score load determined by load criteria tests resulted in tooth breakage with no evidence of galling or pitting of the gear teeth. Failure by tooth breakage appeared independent of the lubricant or temperature. Load criteria tests at pressures simulating altitude conditions resulted in reduced load carrying ability of the lubricants. It appeared deaeration and boiling of the lubricants at these low pressures was the primary reason for the reduced load carrying ability.

ASD TR 61-47

May 1961

SUBJECT: LUBRICATION BEHAVIOR OF LIQUID METALS
INVESTIGATOR: P. H. McDonald, J. K. Whitfield
CONTRACT: AF33(616)-5885, North Carolina State College

ABSTRACT: The analysis presented in this report for a finite length full-journal bearing is based on the hydrodynamic theory of lubrication developed by Reynolds (1886). A finite-difference relaxation procedure is obtained by machine computation. Solutions are obtained for the pressure distribution over the bearing area for various eccentricities in the hydrodynamic range. From the numerical results a simplified journal-bearing design criterion is established. This report surveys the progress in the check out and assembly of the hydrodynamic lubrication test apparatus. A discussion of the apparatus designed and built to study the boundary lubrication properties of liquid metals is given.

WADD TR 61-68, Part I

May 1961

SUBJECT: BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP GREASES FOR HIGH TEMPERATURE APPLICATIONS
INVESTIGATOR: J. J. Chessick
CONTRACT: AF33(616)-7120, Lehigh University
ABSTRACT: Major interest centered on the preparation of thickener solids with improved properties for high temperature applications. Surface treatments of available, high area solids such as silicas and attapulgite clay by phenol-formaldehyde resins or by ion-incorporation techniques proved successful.

WADC TR 53-373 Sup 9

147

WADD TR 61-68, Part I (Continued)

The properties of greases prepared with other thickeners were investigated also. These thickeners included carbon blacks, fiber glass and a finely-divided laboratory prepared MoS₂. Most show promise for use in non-soap grease systems.

Several papers were prepared and submitted for publication. A literature survey of inorganic liquids was undertaken.

WADD TR 61-77

SUBJECT: PROPELLANT LUBRICATION PROPERTIES INVESTIGATION
INVESTIGATOR: M. F. Butner
CONTRACT: AF33(616)-6960, Rocketdyne
ABSTRACT: This report presents the results of friction and wear, ball bearing, and gear tests using RP-1, Ethylene Diamine, Unsymmetrical Dimethyl Hydrazine, Hydrazine, Liquid Hydrogen, Liquid Oxygen, Nitrogen Tetroxide, and Inhibited Red Fuming Nitric Acid as lubricants.

ASD TR 61-144

September 1961

SUBJECT: ACCELERATED STORAGE STABILITY TESTS
INVESTIGATOR: E. N. Cart
ABSTRACT: The storage life of MIL-L-7808 oils has been improved by the use of amine type additives. This report describes the accelerated storage tests used to arrive at qualification limits. The time-temperature relationships for the storage life of MIL-L-7808 oils are given. From these relationships, the storage life can be estimated at any temperature from data at one given temperature.

WADD TR 61-161

May 1961

SUBJECT: RESEARCH ON LUBRICATION BEHAVIOR UNDER EXTREME LOWTEMPERATURES
INVESTIGATOR: R. A. Burton, P. M. Ku
CONTRACT: AF33(616)-7148, Southwest Research Inst.
ABSTRACT: This report is aimed at acquiring a basic understanding of the mechanisms of lubrication, friction and wear at cryogenic temperatures. Work during this period comprised: (1) Development of an apparatus adaptable to

WADD TR 61-161 (Continued)

measurements of friction, wear and related factors down to temperatures as low as -400°F , (2) Development of methods for providing a test environment as free of condensible vapors and chemically active components as practicable, (3) Development of specimen preparation techniques to provide metal surfaces as free of initial contaminant as practicable, (4) Conduction of friction tests on Cu, Au and Fe from ambient to cryogenic temperatures to provide preliminary experimental data, and to aid in development of experimental procedures. On the basis of the above work, it is concluded that the apparatus and techniques are suitable for basic measurements. Examination of the preliminary data shows little friction variation between ambient temperature and -320°F . For Cu vs Cu, a possibly significant drop in friction has been found below -400°F .

ASD TR 61-161, Part II

November 1961

SUBJECT: RESEARCH ON LUBRICATION BEHAVIOR UNDER EXTREME LOW TEMPERATURES. PART II. Clean Metals, Oxide Films, and Organic Films
INVESTIGATOR: J. A. Russell, R. A. Burton, P. M. Ku
CONTRACT: AF33(616)-7148, Southwest Research Inst.
ABSTRACT: Friction measurements at room and cryogenic temperatures are reported for a number of clean metals, oxidized copper, and metals coated with stearic acid monolayers and thin films of selected lubricants. Principal findings were:

(1) For "clean" metal pairs, both like and unlike, friction remained constant from 72 to -424°F .

(2) For oxidized copper pairs, minimal values of friction tended to decrease almost linearly with increasing oxide thickness to about 2500 \AA , remaining constant thereafter. If these minimal values are taken as the lower bound, the data reported show a distinct departure from this lower bound in the 1000 to 2500 \AA range. Such anomalous behavior is tentatively attributed to statistical variation in the composition and mechanical properties of the oxides. Also, a cryogenic effect on the lubricating properties of oxide films was evidenced by the rise in friction for comparable test runs with decreasing temperature.
(3) Friction on monomolecular films of stearic acid on two substrate materials increased from 75 to -320°F . In the case of a film-lubricating a steel pair, friction was shown

ASD TR 61-161, Part II (Continued)

to remain constant from 75 to -25°F, rising linearly to almost double its original value at -200°F and remaining constant at this higher value thereafter to -320°F. Also, the variety of riders and loads employed reflected the extreme strength and endurance of films of this type. (4) Many thin lubricant films exhibited somewhat the same frictional properties as monolayers, with friction rising as temperature decreased. A marked transition to stick-slip friction was noted for DC-510 and SP4E oils at temperatures above -20°F.

ASD TR 61-301

September 1961

SUBJECT: FRICTION AND WEAR CHARACTERISTICS OF CERMETS AT HIGH TEMPERATURE AND HIGH VACUUM

INVESTIGATOR: R. D. Brown, R. A. Burton, P. M. Ku

CONTRACT: AF33(616)-7209, Southwest Research Inst.

ABSTRACT: The friction and wear characteristics of several bearing substrate materials and lubricants were determined over a temperature range from ambient to 2000°F, and in normal atmosphere with pressure varied from 760 to 10⁻⁶ mm Hg. The sliding speed was 14 to 20 fpm. The load ranged from 1,500 to 15,000 psi. Two oxide-bonded cermets (Cr-Al₂O₃-W and Cr-Al₂O₃-Mo-TiO₂) were found to exhibit low wear and moderate friction when run against themselves throughout a wide range of test conditions. A third cermet (Cr-Al₂O₃) showed very poor frictional properties. Three high-temperature alloys were also investigated for purposes of comparison. Test results are also presented which indicate that two of the cermets were effectively lubricated by precious metal films.

ASD TR 61-459

January 1962

SUBJECT: LUBRICATION BEHAVIOR OF LIQUID METALS

INVESTIGATOR: P. H. McDonald, J. P. Lamb

CONTRACT: AF33(616)-5885, North Carolina State College

ABSTRACT: This report surveys recent progress in the preliminary testing of experimental equipment for investigating the hydrodynamic and boundary lubrication behavior of liquid metals. Results of the first boundary lubrication tests are presented and briefly discussed.

ASD TR 61-500

January 1962

SUBJECT: MECHANISMS OF FRICTION AND WEAR BETWEEN SOLID SURFACES
INVESTIGATOR: K. E. Boyd, C. T. Rollins, A. D. Thomas
CONTRACT: AF33(616)-6833, University of Utah
ABSTRACT: A low-velocity friction testing machine for surface velocities of from 1.0 foot per second to 15.0 feet per second and a high-velocity friction testing machine for surface velocities of from 1.0 foot per second to 200 feet per second are designed, fabricated and used to test a number of pairs of solid materials. Coefficients of friction as a function of several parameters were measured and plotted. It was found that a unique interface temperature between rubbing materials does not exist but rather a random extremely variable temperature profile of considerable magnitude does exist. Data was obtained which gives evidence for deducing the effects of reactivity and solid solubility on the friction process.

ASD TR 61-652

March 1962

SUBJECT: DEVELOPMENT OF A PROTOTYPE RADIATION RESISTANT BEARING AND GEAR LUBRICANT
INVESTIGATOR: C. L. Mahoney
CONTRACT: AF33(616)-7601, Shell Development Co.
ABSTRACT: The polyphenyl ethers have the physical properties and stability to permit their use under environments far too severe for conventional lubricants. Their lubricating properties are much better than other stable viscosities. While their high-speed bearing performance is good, viscosities at high temperatures are too low to give sufficient load carrying capacity for some gear applications.

In this work, additives have been examined as a means of improving their gear load-carrying capacity.

Suitable additives must meet the conflicting requirements of high activity towards metal surfaces and low effect on the stability of the ethers. No additive examined completely satisfied these requirements. Only aromatic derivatives had sufficient stability and these materials generally did not improve load carrying capacity. However, some aromatic phosphate derivatives, when used in high concentrations (3-10%), appreciably improved the lubrication properties, and their effects on the oxidation, radiation and thermal stability of the polyphenyl ethers was low.

ASD TR 61-652 (Continued)

Such additives may be suitable where requirements are low.

ASD TR 61-729

February 1962

SUBJECT: INORGANIC THICKENED GREASES
INVESTIGATOR: J. B. Christian
ABSTRACT: Experimental non-soap-synthetic base greases have been synthesized and studied utilizing standard and specialized techniques. The greases were synthesized from several types of base fluids including silicones, polyphenyl ethers, silphenylenes and/or blends of these materials. New and improved inorganic and organic thickeners were incorporated in the formulations. Several of the grease formulations show encouraging promise for such applications as anti-friction bearing lubricants, high temperature pneumatic greases, and lubricants for sliding and rolling friction under extremely heavy load conditions.

ASD TR 61-737

January 1962

SUBJECT: MATERIALS RESEARCH FOR LUBRICANTS AND HEAT TRANSFER FLUIDS
INVESTIGATOR: K. R. Mecklenburg
CONTRACT: AF33(616)-6854, Midwest Research Inst.
ABSTRACT: Friction data are presented for copper on copper and for titanium on nickel for very low sliding speeds and light loads. The stick-slip apparatus used to obtain these data is described. Results are given for the initial part of an investigation in which the film conductance will be determined for liquid metals. A preprototype liquid metal boiler was operated at temperatures up to 1630°F with sodium. The design of a prototype liquid metal boiler for use in the next step of this work at temperatures up to 1800°F is described. Techniques used to handle sodium are outlined. Bearing life data are included for experimental greases run at high temperatures and speeds in the Pope Spindle. Screening runs with experimental grease made on the Navy Spindle at 350°F were not effective in selecting greases which would consistently run more than a few hours at high temperatures in the Pope Spindle. Wear scar data from the Four-Ball Wear Tester are given for experimental fluids.

SOLID FILM LUBRICANTS

WADD TR 60-530, Part II

April 1961

SUBJECT: CERAMIC BONDED SOLID-FILM LUBRICANTS
INVESTIGATOR: M. T. Lavik
CONTRACT: AF33(616)-6115, Midwest Research Institute
ABSTRACT: Films of PbS + MoS₂/B₂O₃ were studied in air and vacuum (1×10^{-5} mm. Hg) from 120° to 1000°F. Friction coefficients in air are similar to those in vacuum. Film wear-life is significantly higher in vacuum. The friction of some bonded pellets (PbS/B₂O₃) is similar to bonded films of the same compositions. Friction data of several lubricant pellets bonded with Na₂B₄O₇ and Na₃PO₄ suggest new film formulations for study. Differential thermal analysis has been used to measure the temperature at which endothermic and exothermic reactions take place in several solid lubricants. A vacuum friction machine was fabricated for studying lubricant films in the temperature range of 100° to 1500°F and pressures ranging from 760 to 1×10^{-5} mm. Hg.

WADD TR 60-530, Part III

February 1962

SUBJECT: PHYSICAL AND CHEMICAL PROPERTIES OF CERAMIC BONDED SOLID LUBRICANT FILMS
INVESTIGATOR: M. T. Lavik, B. Daniel, T. Medved
CONTRACT: AF33(616)-6854, Midwest ResearchInst.
ABSTRACT: The performance, structure, and surface morphology of PbS:MoS₂:B₂O₃ lubricant films have been investigated. Wear-lives measured in air and vacuum from 500 to 1250°F were dependent on baking procedure and substrate material. Electron micrography was used to: (1) determine the particle size distribution of the film components (a majority of both PbS and MoS₂ particles are less than 0.6 micron in diameter), and (2) study film failure and wear particle formation on the rubbed surfaces. Unexpected crack patterns were observed which may be related to wear particle formation. The effects of heat on phase stability and chemical reactivity of the film and its components were studied using DTA, TGA, and IR absorption. Shear and longitudinal sonic wave velocities were measured in MoS₂:B₂O₃ by a pulse-echo technique, $V_s = 1.26 (\pm 0.02) \times 10^5$ cm/sec; $V_L = 1.83 (\pm 0.02) \times 10^5$ cm/sec.

WADD TR 60-823

November 1960

SUBJECT: SOLID FILM LUBRICANTS FOR HIGH TEMPERATURE
NUCLEAR ENVIRONMENTS

INVESTIGATOR: B. Daniel

CONTRACT: AF33(616)-6728, Midwest Research Inst.

ABSTRACT: Ten compounds which showed promise as solid lubricants in pellet friction experiments were chosen for a study of the effects of neutron and gamma radiation on their solid lubricant properties. Three static irradiation programs were undertaken: (1) mixed reactor flux to 10^{16} nvt fast neutrons; (2) mixed reactor flux to 10^{20} erg/g C dose. All of the irradiation had been accomplished by the end of the first year of the project. A new friction test machine was designed and constructed for evaluating lubricant films operating in a gamma ray flux. The films, coated on spherical bearings, are tested in oscillatory motion under loads to 10,000 lb., from room temperature to 1000°F. This Oscillatory Spherical Bearing Tester (MRI Friction Test Machine Mark VII) was completed, checked out, and ready for operation at the Southwest Research Institute gamma irradiation facility at the end of the project's first year. The frictional behavior of composite lubricant-binder pellets was studied over a temperature range 80-1000°F. Twenty-two different composites were tested. MoS₂ bonded with Na₂B₄O₇ or Na₃PO₄ shows good lubricity over the entire temperature range.

WADD TR 60-823, Part II

July 1961

SUBJECT: SOLID FILM LUBRICANTS FOR HIGH TEMPERATURE
NUCLEAR ENVIRONMENTS

INVESTIGATOR: B. Daniel

CONTRACT: AF33(616)-6728, Midwest Research Inst.

ABSTRACT: A gamma radiation dose of 0.878×10^{12} erg/gC had no effect measurable within experimental variation on the wear-life of PbO bonded with B₂O₃ and MoS₂ bonded with NaOSiO₂. A new friction machine was designed and constructed for evaluating solid films operating in a gamma flux. The effect of gamma doses $(21.6 \text{ to } 41.8) \times 10^8$ erg/gC on the wear-life of MoS₂ bonded with Na₂B₄O₇ and Lubricant A could not be measured within experimental variation. The effect of gamma and neutron radiation on lattice spacings of solid lubricant pigments was studied by X-ray powder patterns. A program of electron microscope observations of the radiation effects on dislocations in solid lubricants was begun.

WADD TR 61-49, Part I

March 1961

SUBJECT: A STATISTICAL ANALYSIS OF THE FRICTIONAL
PERFORMANCE OF SOLID FILM LUBRICANTS
PART I. Resin Bonded Films in Air

INVESTIGATOR: M. R. Adams

ABSTRACT: This report presents an analysis of variance to establish the statistical significance of load and sliding speed in determining wear life values for nine commercially available organic bonded dry film lubricants. Experiments were conducted on the Hohman A-6 tester with two loading shoes. Statistical calculations were made to show that the correlation between the A-6 and the standard Alpha tester with respect to wear life is only slight. General equations for the analysis of variance in a 2^2 factorial experiment were derived. These equations are particularly useful for analyzing the non-orthogonal case with unequal numbers of replications at each of the four combinations of variables.

ASD TR 61-222

September 1961

SUBJECT: SURVEY OF SOLID FILM LUBRICANTS

INVESTIGATOR: B. D. McConnell

ABSTRACT: A survey of the state-of-the-art in the field of solid film lubrication is presented. Presently available solid film lubricants are discussed as to their uses and limitations. The data presented indicate these films are capable of operation up to approximately 500°F in slow speed sliding moderately loaded bearing applications. The limiting factor is the epoxy or phenolic resin normally used as a binder for these films. Preparation of these films as well as various types of equipment for studying friction, wear and life of the films are discussed.

Present research to obtain solid film lubricants capable of operating at 1000°F or higher is discussed. The results obtained to date indicate a metal salt-ceramic binder formulation as the most promising approach. A lead sulphide (Pbs)-Boric Oxide (B_2O_3) combination has shown the best lubrication characteristics for use at 1000°F, but suffers short life and high friction and wear below 800°F. Other metal salt-ceramic binder combinations show the same trend of poor lubrication characteristics at the lower temperatures. Methods of combating this deficiency are presented.

ASD TR 61-222 (Continued)

Future requirements are briefly discussed along with currently planned programs to extend the operating capability to 1500°F or higher and to include vacuum as well as oxidizing environments.

ASD TR 61-695

March 1962

SUBJECT: EXACT HEAT EQUATION SOLUTIONS REVELANT TO THE MEASUREMENT OF JUNCTURE CONDITIONS AT THE INTERFACE OF MOVING CONTACTS

INVESTIGATOR: F. F. Ling, C. W. Ng

CONTRACT: AF33(616)-8016, Rensselaer Polytechnic Institute

ABSTRACT: Exact solutions of the heat equations in two dimensions are obtained for: a rotating disc, insulated on the flat surface, with an arbitrary heat input distribution over a circular arc, fixed in space, along the periphery of the disc while the rest of the periphery, not receiving heat at any given moment, radiates into the ambient; and a truncated circular sector (fan shaped), insulated on four flat surfaces, with an arbitrary heat input distribution over the truncated, curved side and radiates into a controlled ambient. The solution made possible the indirect measurement of point-wise juncture condition at the interface of sliding contacts; the actual mechanical model is only described schematically. The solutions are of such forms as to be most suitable for matching the solutions at the interface, given by juncture condition.

ASD TDR 62-55

January 1962

SUBJECT: LUBRICATION STUDIES WITH LAMELLAR SOLIDS

INVESTIGATOR: P. Bryant

CONTRACT: AF33(616)-7823, Midwest Research Inst.

ABSTRACT: A basic research program is being conducted to determine the mechanisms of friction and wear for lamellar solid lubricants. Single crystals of graphite were grown and an UHV (2×10^{-13} Torr) controlled atmosphere system was perfected. A stress-etch mechanism is proposed here to explain the effect of atmospheric gases upon the lubrication properties of lamellar solids. The proposed mechanism describes the observed reduction of cohesive energy (mica was 30 times stronger in vacuum than in air) by an external attack upon the bifurcation line or shearing edge; thus depending on the well established processes of surface adsorption and migration without requiring diffusion of air molecules between lamellae.

ELECTRICAL & ELECTRONIC MATERIALS

WADC TR 59-337, Part II, OTS Release

September 1960

SUBJECT: SYNTHESIS AND PURIFICATION OF DIELECTRIC MATERIALS
INVESTIGATOR: W. C. Divens, D. H. Hogle, D. W. Lewis, P. A. Tierney, T. W. Dakin, D. Berg
CONTRACT: AF33(616)-5979, Westinghouse Electric Co.
ABSTRACT: This report describes the progress during the second year on a research program undertaken to prepare pure dielectrics with improved properties for use as electrical insulation at 500°C. The effort has been concentrated on the materials: boron nitride, alumina, and boron phosphide.

Boron nitride has been prepared with better dielectric properties, at 500°C, than any other report materials. The condition for the hot pressing of the boron nitride discs have been elucidated. Hot pressed boron nitride has been shown to possess electrical properties which are anisotropic.

Anodized aluminum oxide films have good 500°C dielectric properties. The films are polarity sensitive and have a resistance which decreases with increasing field.

The adhesion of the electrode metal to the anodized oxide film has been improved appreciably by first evaporating silicon monoxide in vacuum, and then evaporating gold on top of the silicon monoxide without having released the vacuum.

Dielectric measurements of aluminum oxide films formed by hydrolysis of aluminum isopropoxide have indicated inferior properties for the oxide made in this manner.

Boron phosphide has been synthesized by two means: (1) direct reaction of the elements, boron and phosphorus, and (2) by reaction of boron trichloride, phosphorus and hydrogen.

WADC TR 59-337, Part III

June 1961

SUBJECT: SYNTHESIS AND PURIFICATION OF DIELECTRIC MATERIALS
INVESTIGATOR: W. C. Divens, D. H. Hogle, D. W. Lewis, P. A. Tierney, T. W. Dakin, D. Berg

WADC TR 53-373 Sup 9

157

WADC TR 59-337, Part III (Continued)

CONTRACT: AF33(616)-5979, Westinghouse Electric Corp.
ABSTRACT: This is the final report on a contract to synthesize and purify inorganic dielectrics to achieve better dielectric properties at high temperatures up to 500°C. The research program has concentrated particularly on boron nitride and aluminum oxide, with some work on several other materials. During the last year, effort has concentrated on preparing thin films of these materials and evaluating their dielectric properties at high temperature. Thin films of boron nitride and anodically formed alumina have been developed with quite satisfactory dielectric properties at 500°C. Work on arc plasma jet sprayed inorganic films starting with high purity materials has yielded only films with inferior dielectric properties, indicating contamination from the arc electrodes.

WADD TR 60-56, Part IV

December 1961

SUBJECT: A COMPENDIUM OF THE PROPERTIES OF
MATERIALS AT LOW TEMPERATURE (PHASE II)
INVESTIGATOR: R. B. Stewart, V. J. Johnson
CONTRACT: DO33(616)-59-6, National Bureau of
Standards
ABSTRACT: Phase II of the Compendium includes data sheets on compressibility factor, velocity of sound and entropy of fluids, vapor-liquid equilibrium concentration of binary mixtures of fluids, and electrical resistivity and thermal conductivity integrals of metallic solids. Data sheets are included for each of these properties for the following materials: Compressibility Factor (Helium, Hydrogen, Neon, Nitrogen, Air, Methane); Entropy (a T-S diagram for Neon); Velocity of Sound (in liquids: Helium, Hydrogen, Nitrogen, Oxygen, Argon, Methane; in gases: Helium, Hydrogen, Neon, Nitrogen, Oxygen, Air, Carbon Monoxide, Argon, Methane); Liquid-Vapor Equilibrium Concentrations (Helium in Hydrogen, Nitrogen, Methane; Hydrogen in Nitrogen, Carbon Monoxide, Methane; Nitrogen in Oxygen, Carbon Monoxide, Argon, Methane); Electrical Resistivity (53 of the pure metallic elements); and Thermal Conductivity Integrals (44 pure metallic substances, 36 non-ferrous alloys, 9 ferrous alloys and 4 glasses and plastics). In general, the data sheets present the data primarily in graphical form, and in addition include tables of selected values, references to the sources of the data and other references. Appropriate comments of interest to the user are also given.

June 1961

SUBJECT: SUPERCONDUCTIVITY IN METALS AND ALLOYS
INVESTIGATOR: W. H. Cherry, G. D. Cody, J. I. Gittleman
CONTRACT: AF33(616)-6405, RCA Laboratories
ABSTRACT: A proposed examination of the thermal contact resistance (Kapitza resistance) between metals and liquid helium is described. The apparatus is designed to measure changes in this contact resistance at the superconducting transition as well as in the normal state. Measurements of the effect of hydrogen and deuterium on the superconducting transition temperature; hydrogen depresses the transition temperature more than deuterium. Measurements have been made of the transition temperatures in the system (Nb, Ta, V)₃Sn. The transition temperatures range from 2.8°K to 18°K and can be related to a simple mass and volume dependence. Critical field measurements indicate behavior similar to that in other "hard" superconductors. Resistance measurements have revealed a resistance anomaly near 100°K which can be related to an existing theory. It has been found possible to prepare films of Nb₃Sn by a transport reaction. Alloying experiments indicate ambiguities in the electron-to-atom ratio ascribed to various elements.

Methods have been developed for the measurement of the interphase energy in superconductors. A description of the method is given along with preliminary results on thin foils and films of tin.

January 1961

SUBJECT: INVESTIGATION OF THE STOICHIOMETRIC AND STRUCTURAL VARIABLES WHICH AFFECT CONDUCTIVITY IN FERROMAGNETIC CERAMIC MATERIALS FOR USE IN MAGNETIC CIRCUITS
INVESTIGATOR: F. H. Horn
CONTRACT: AF33(616)-6499, General Electric Co.
ABSTRACT: This report deals with the development of the Czochralski method of growing single crystal iron ferrites of the high purity and crystalline soundness necessary to make progress in studies directed toward understanding the electrical properties of ferrites.

Only reflectivity peaks have been observed from studies of the optical reflectivity and transmission of magnetite. Resistivity and thermo-electric power as a function of temperature are reported for magnetite. Hall effect studies are inconclusive. Discrepancies between previous data and those obtained here are noted. The thermal

WADD TR 60-421, (Continued)

conductivity of magnetite has been measured as a function of temperature. The room temperature value is about 70 milliwatts, and an abrupt change is noted at the 120°K transition.

WADD TR 60-608, OTS Release

January 1961

SUBJECT: STUDIES OF TRANSIENT HEAT CONDUCTION AT HIGH THERMAL FLUX
INVESTIGATOR: H. N. Abramson, W. Chu, J. C. Cook
CONTRACT: AF33(616)-6323, Southwest Research Inst.
ABSTRACT: This report presents the results of a theoretical and experimental study of transient heat conduction in cylindrical specimens of copper, Bakelite, stainless steel and graphite, subjected to an incident thermal flux of approximately 500 cal/cm²-sec. Theoretical investigations included the solution of the transient heat flow equation for cylindrical rods of finite length and with temperature dependent thermophysical properties. Comparisons of this theory, and similar theoretical approaches by other investigators, with experimental results show a large and consistent difference for all materials investigated. Although the experimental results are preliminary in nature and require further confirmation, the results obtained thus far indicate a serious inadequacy of present heat transfer theory for thermal fluxes of this order of magnitude, and the consistency of these results suggests that a considerably modified theory must be developed to achieve qualitative and quantitative agreement with experiment.

WADD TR 60-787, OTS Release

July 1961

SUBJECT: INVESTIGATION OF THE STRUCTURAL AND MAGNETIC PROPERTIES OF THIN FERROMAGNETIC FILMS
INVESTIGATOR: W. D. Doyle, J. Johnson, J. E. Rudisill, F. R. L. Schoning, H. G. F. Wilsdorf, A. E. Berkowitz
CONTRACT: AF33(616)-6298, The Franklin Institute
ABSTRACT: The principle object of the research is the correlation of the magnetic and structural properties of thin ferromagnetic films. This report, which describes in detail the instrumentation, the film preparation and the first results obtained with 77:23 permalloy films, covers the first phase of the work. The structural investigation

WADD TR 60-787 (Continued)

was carried out by using electron microscopy and electron diffraction techniques. Replicas of the surface of the films indicated the presence of a roughness which, at present however, has not been correlated to the magnetic uniaxial anisotropy of the films. The magnetic experiments included the recording of hysteresis loops with an ac hysteresigraph, the observation of domain patterns in a Kerr Magneto-Optic apparatus and the measurement of the angular dependence of the torque with a sensitive torque magnetometer. The magnetic data are discussed in terms of the behavior of single domain and multi domain magnetic particles.

WADD TR 60-899, Part II

July 1961

SUBJECT: THE DEVELOPMENT OF SILICA HOLLOW MICRO-SPHERES FOR USE AS A HIGH TEMPERATURE DIELECTRIC

INVESTIGATOR: J. W. Leforge, R. S. Lothrop

CONTRACT: AF33(616)-7263, Emerson & Cuming, Inc.

ABSTRACT: Silica microbubble material has been produced by melting particles of silica and expanding them into bubbles at 1700°C and by acid-leaching of the 16% sodium glass bubbles which are made commercially at 950°C, followed by dehydration at 1140°C. Both materials were bonded into sheet structures using cold set cement type bonds without serious degradation of the dielectric properties. These structures withstand moderate loads at 1100°C and do not collapse at 1300°C. Densities range from 0.6 to 1.2 g/cc.

WADD TR 60-909

April 1961

SUBJECT: SYNTHESIS OF NEW DI-ELECTRIC MATERIALS BY ISOMORPHOUS SUBSTITUTION FOR SILICON IN FLUOR-PHLOGOPITE

INVESTIGATOR: E. A. Schatz, J. C. Withers

CONTRACT: AF33(616)-6827, American Machine & Foundry Company

ABSTRACT: Attempts to substitute for silicon in the fluor-phlogopite structure have resulted in the synthesis of a fluor-germanium mica and a boron phosphate mica-like orthorhombic crystal. The batch material contained Al_2O_3 , MgO , MgF_2 , K_2CO_3 , plus GeO_2 for forming the fluor-germanium mica and BPO_4 for forming the boron phosphate crystal. Optical analysis with a polarizing microscope and by X-ray diffraction verified the mica structure of the germanium crystal and

WADD TR 60-909 (Continued)

disproved the mica structure of the boron phosphate crystal. The indices of refraction and the unit cell constants were determined for both crystals. A study of the electrical properties up to temperatures of 1000°F showed that the dielectric properties of the fluor-germanium mica are very similar to that of fluor-phlogopite.

ASD TN 61-32, OTS Release

May 1961

SUBJECT: A LABORATORY ARC MELTING FURNACE FOR THE PRODUCTION OF ALLOY SAMPLES FROM RE-ACTIVE METALS

INVESTIGATOR: J. C. Olson

ABSTRACT: An arc melting furnace for the production of small samples of alloys and intermetallic compounds has been designed and built. It is used in phase diagram investigations involving reactive materials such as rare earth metals. It has also been used for the laboratory scale preparation of nickel-copper alloys being studied for magnetic thermometry.

In this report, the design, capabilities and operation of the furnace are described.

WADD TR 61-90, OTS Release

July 1961

SUBJECT: INVESTIGATION OF THE APPLICATION OF ELECTRIC CURRENT DURING ZONE REFINING

INVESTIGATOR: L. L. Thomas

CONTRACT: AF33(616)-6150, Research Chemicals

ABSTRACT: Continuation of research on the application of electric current during zone refining of germanium. Modified zone refining equipment and resistivity profile measuring apparatus are described. Results, presented graphically, indicate that segregation rate is increased when the zone travels from the negative end to the positive end of the ingot and segregation is suppressed when the electric current is reversed. Theoretical analysis indicates that the observed segregation rate is much larger than that predicted on the basis of ion migrations in an electric field.

ASD TN 61-160, OTS Release

February 1962

SUBJECT: STATE-OF-THE-ART SURVEY OF SUPER CONDUCTIVITY OF SUPERCONDUCTING ELECTRO-MAGNETS

INVESTIGATOR: R. A. Wolf

ABSTRACT: Some of the important characteristics and applications of superconducting materials have been compiled from a survey of existing information on the subject of superconductivity. The basic properties of superconductors are described and a brief account of the Bardeen-Cooper-Shrieffer theory is given.

The previously published data on the superconducting compound Nb_3Sn (niobium-tin) is reviewed, and superconducting solenoids utilizing this material are discussed.

ASD TR 61-273, OTS Release

November 1961

SUBJECT: CONSTRUCTION AND CALIBRATION OF A HALL-EFFECT GAUSSMETER

INVESTIGATOR: K. Strnat, R. A. Wolfe

ABSTRACT: A gaussmeter, based on the Hall effect, was built for laboratory use. It permits easy and quick measurement of steady magnetic fields in ten ranges between 1 gauss and 30 kilogauss. The small indium-arsenide probes facilitate the mapping of non-uniform fields and provide access to the interior of small coils and into narrow gaps. The report describes the meter design and calibration.

ASD TR 61-498, OTS Release

November 1961

SUBJECT: THE CRYSTAL LATTICES IN ALNICO PERMANENT MAGNETS STUDIED BY TRANSMISSION ELECTRON DIFFRACTION

INVESTIGATOR: K. J. Kronenberg

CONTRACT: AF33(616)-7287, Indiana Steel Products Div.

ABSTRACT: Alnico permanent magnet materials have been investigated with transmission electron metallography. A method was developed for producing samples of these brittle alloys which were thin enough for the transmission of 80 KV-electrons.

The precipitation process of Alnico V was studied in the material itself by transmission electron diffraction. The unusual development of a precipitate network

ASD TR 61-498 (Continued)

found earlier (WADD TR 58-536) by less immediate replica methods was confirmed. The cast material has a bcc crystal lattice with a pronounced super-structure. After complete heat treatment a precipitated phase is found predominant in Alnico V with a Bcc crystal lattice which forms a sort of single crystal with a mosaic structure.

The diffraction patterns of various technically important Alnico materials have been compared. The cast alloys have crystal lattices with similar degrees of order and superstructure. Their lattice parameters vary with the compositions of the alloys between 3.16A and 2.80A.

After their heat treatments a precipitated phase with a bcc lattice is found in all these alloys. The precipitates in the different alloys are similar in composition since they all have the same lattice parameter of 2.88A. The degree of order obtained during the heat treatment is very different for the different alloys. The magnetic properties of the Alnicos are related to the degrees of order determined by the appearance of the diffraction patterns.

An analysis of the phases attempted earlier was confirmed by diffraction study.

ASD TR 61-630, OTS Release

February 1962

SUBJECT: RESEARCH ON SPONTANEOUS MAGNETIZATION IN SOLID BODIES
INVESTIGATOR: I. S. Jacobs, D. S. Rodbell, W. L. Roth
CONTRACT: af33(616)-7396, General Electric
ABSTRACT: A number of research projects dealing with fundamental interactions and the microstructure of internal fields in selected magnetic materials were carried out. (a) The nuclear magnetic resonance of Co^{59} in metallic cobalt powders has been examined. In addition to the absorption characteristic of Co^{59} in face-centered cubic cobalt, there were found an additional group of absorptions, one of which is clearly associated with hexagonal close packed cobalt. (b) The phenomenon of spin-flopping in antiferromagnetic MnF_2 , in which the axis of antiferromagnetism is decoupled from the crystal axes, was observed by magnetization measurements in high pulsed magnetic fields. Its position coincided with the value, 93 kOe, predicted from measurements of antiferromagnetic resonance with millimeter microwaves. (c) The temperature dependence of the magnetocrystalline

ASD TR 61-630 (Continued)

resonance on single crystal thin films and submicron precipitate particles in Cu, at temperatures below the range in which this is the stable structure. Satisfactory agreement is obtained for a tenth power law between anisotropy and magnetization. (d) A very brief review paper on pulsed field magnetization measurements in compounds was prepared. (e) An investigation of magnetic exchange and structure in lanthanum manganite perovskite compounds spanning the transition between antiferromagnetism and ferromagnetism was performed using neutron diffraction and high field magnetization techniques. Evidence favored the single-phase canted spin model for this region over the model of a mixture of two crystallographically similar but magnetically distinct phases. (f) Seven additional exploratory projects were undertaken, some of which hold considerable promise for development.

ASD TR 61-693

March 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON CORONA-RESISTANT MATERIALS
INVESTIGATOR: J. C. Devins, H. L. Greenhaus, D. R. Johnson, F. Y. DuPont, A. L. Lynn, A. Pletenik, H. G. Pfeiffer
CONTRACT: AF33(616)-7485, General Electric Research Laboratory
ABSTRACT: The shapes of individual corona pulses and the changes in the shape with the applied voltage were determined. It was found that as the overvoltage increased the pulses change from a type characteristic of corona avalanches to one characteristic of streamer discharge. The change has been interpreted theoretically by consideration of the positive ion space charge produced in the early stages of the discharge. The chemical reactions have been studied in detail on polyethylene. On other insulation materials the gross reactions have been studied with an eye to a broad general method of testing.

The work done in Phase II of the contract was in three general areas:

A. Measurement of corona pulse size distributions in cylindrical corona cells, power measurements, and theoretical considerations.

B. Assessment of physical and chemical

ASD TR 61-693 (Continued)

damage as a result of cylindrical cell corona exposure on Mylar, nylon, and a Formvar varnish.

C. Determination of damage to Mylar under various conditions of divergent field corona. Charge profiles and erosion are included.

NUCLEAR MEASUREMENTS

WADD TR 60-576, OTS Release

December 1960

SUBJECT: PHOTOCONDUCTIVITY IN CdS CRYSTALS AS A MECHANISM FOR GAMMA RAY DOSIMETRY
INVESTIGATOR: O. V. Sessoms
ABSTRACT: Gamma ray induced changes in the conductivity of CdS crystals are studied as a possible mechanism for monitoring gamma dose rates. Data is presented on the change in conductivity due to gamma exposure over a range of 1.4×10^5 ergs/g hr (C) to 5×10^7 ergs/g hr (C). The change in photocurrent as a function of voltage at two dose rates is also reported. The rise time of this photocurrent is investigated for photons in the visible light range as well as for Co 60 gamma rays.

ASD TR 61-32

August 1961

SUBJECT: FURTHER DEVELOPMENT OF MICROWAVE GAMMA-RAY ION CHAMBER
INVESTIGATOR: J. Aichroth, R. Slevin, K. Speh
CONTRACT: AF33(616)-6971, Cutler-Hammer, Inc.
ABSTRACT: A microwave gamma-ray ion chamber has been developed that measures gamma radiation from 10^6 to 10^{10} ergs per gram-hour (carbon) in the presence of a neutron flux of 10^{10} neutrons per square cm per second. This report discusses the choice of materials, development of a unique vacuum system, and fabrication of chambers suitable for operation at temperatures to 500°C in a high neutron flux. Data are presented on the response of an ion chamber to various ratios of incident gamma, thermal neutron, and fast neutron fluxes.

ASD TR 61-169, OTS Release

June 1961

SUBJECT: DOSE RATE EFFECTS IN LIQUID HYDROCARBON RADIOLYSIS
INVESTIGATOR: T. D. Nevitt, W. A. Wilson
CONTRACT: AF33(616)-7089, American Oil Co.
ABSTRACT: Radiolysis of cyclohexane, hexane and pentane were examined at dose rates from 2×10^{16} , to 5×10^{24} ev-g-sec. Cyclohexane shows no variation of products over this dose-rate range. Small changes may occur in the low molecular weight products from hexane. Ratios of C_{12} hexane products and C_{10} pentane products show small but

WADC TR 53-373 Sup 9

167

ASD TR 61-169 (Continued)

definite dose rate dependence. Results indicate a more marked dependence on dose rate might be found at dose rates lower than examined here.

THERMO PHYSICS

WADD TR 59-366, Part III, OTS Release

July 1961

SUBJECT: THERMAL PROTECTION OF STRUCTURAL,
PROPULSION, AND TEMPERATURE-SENSITIVE
MATERIALS FOR HYPERSONIC AND SPACE FLIGHT.
PART III. Analytical Studies of Phenomena
for Thermal Protection

INVESTIGATOR: W. E. Manos, D. E. Taylor

CONTRACT: AF33(616)-6006, Laboratories for Applied
Sciences

ABSTRACT: This report presents the results of both
analytical and experimental investigations of the applicability
of the mass transfer cooling phenomenon to the protection of
materials subjected to severe thermal environments. It also
summarizes efforts to improve characterization of the ex-
perimental thermal environment provided by the one-megawatt
air-stabilized arc. These efforts include extended develop-
ment of calorimetry to provide measurement of heat flux rates
at surface temperatures in excess of 3000°F and utilization
of additional techniques for spectroscopic temperature de-
termination.

WADC TR 59-510, Part II

February 1961

SUBJECT: STANDARDIZATION OF THERMAL EMITTANCE
MEASUREMENTS

INVESTIGATOR: W. Harrison, J. Richmond, E. Plyler, R.
Stair, H. Skramstad

CONTRACT: AF33(616)-58-20, National Bureau of
Standards

ABSTRACT: Work during the year was directed toward
establishment of equipment and procedures for the evaluation
of normal spectral emittance of specimens held at tempera-
tures in the range of approximately 800° to 1400°K (980° to
2060°F), over the wavelength range of 1 to 15 microns. Some
of the developments of the previous contract were modified
to increase precision and reduce errors in the measurements.
One such modification was to provide means whereby a reference
blackbody furnace and the hot specimen could be interchanged
at will as sources of radiant flux to be measured relative
to that from the comparison blackbody furnace. The apparent
emittance of the specimen and of the reference blackbody
furnace were both measured relative to the comparison black-
body furnace. The apparent emittance of the specimen at each
of numerous selected wavelengths was then divided by the

WADC TR 59-510, Part II

February 1961

corresponding apparent emittance of the reference blackbody furnace to obtain the reported spectral emittance of the specimen. Assembly of the equipment for determination of spectral reflectance, under conditions approximating normal illumination and hemispherical viewing, was completed.

WADD TR 60-102

March 1961

SUBJECT: INVESTIGATION OF HIGH EMITTANCE COATINGS
TO EXTEND THE MACH NUMBER RANGE OF APPLI-
CATION OF STRUCTURAL MATERIALS

INVESTIGATOR: A. Gravina, M. Katz

CONTRACT: AF33(616)-5925, Republic Aviation Corp.

ABSTRACT: The value of knowing and being able to modify the thermal emittance of structural materials employed in space and missiles work is well known. This report describes the design and assembly of an apparatus capable of measuring the total and spectral normal emittance of solids at temperatures from 400 to 1800°F, and pressures from atmospheric to less than 5 microns Hg. Ceramic, paint, oxide, and metallic coatings applied to Inconel X, A-286 steel, and 6 Al-4V titanium were investigated. Emittance data are presented for materials tested after short term, prolonged, and cyclic temperature exposures in the noted temperature and pressure range of the instrument. The effect of contamination is also considered for some materials. It is shown that the emittance of metal substrates can be appreciably altered by the application of suitable coatings.

WADD TR 60-190, OTS Release

November 1961

SUBJECT: ELECTRON BEAM TECHNIQUES FOR MEASURING
THE THERMOPHYSICAL PROPERTIES OF
MATERIALS

INVESTIGATOR: J. M. Cercao, H. M. Childers

CONTRACT: AF33(616)-6527, American Machine & Foundry
Company

ABSTRACT: The determination of the suitability of electron bombardment for measuring the thermophysical properties of materials is made. A theory is developed which is applicable to the case of a radiating cylindrical sample. Conclusions are that the technique offers a valuable tool for measuring these properties within described limits. Values of α , k , ρc_p and ϵ were determined for graphite and Al₂O₃ (Polycrystalline) and found to be accurate within the

WADD TR 60-190 (Continued)

limitations inherent in the equipment used. Recommendations on instrumentation are made for the further development of this technique.

WADD TN 60-251

June 1961

SUBJECT: ANALYSIS AND DESIGN OF AN ELECTRON GUN-MOLECULAR BEAM INSTRUMENT FOR USE IN FREE RADICAL STUDIES

INVESTIGATOR: Lt M. Charles

ABSTRACT: This technical note shows how an instrument can be developed and built for use in studying primary and intermediate effects of radiation upon different materials. This instrument is an electron gun-molecular beam device. Its function is to form a molecular beam with a long mean free path length from a known gas. The molecular beam is then irradiated by the electron gun, producing excited states, ions, and free radicals. Due to the length of time from irradiation to being collected on a 4.2°K cold finger, the excited states and ions return to their normal state, leaving only the free radicals for study. The free radicals are then studied at different temperatures from 4.2°K to room temperature with the use of infrared (IR) and electron paramagnetic resonance (EPR) techniques. It is believed that this method will produce an accurate, reliable history of radiation effect processes which a material will undergo after initial irradiation.

WADD TN 60-273, OTS Release

July 1961

SUBJECT: RESULTS OF ABLATION TESTS ON SEVERAL PLASTIC MODELS IN A HYPERSONIC WIND TUNNEL.

INVESTIGATOR: C. Economos

CONTRACT: AF33(616)-5944, Polytechnic Institute of Brooklyn

ABSTRACT: An experimental study of transient ablation has been carried out in a facility developed expressly for this purpose. The facility utilizes the PIBAL pebble bed convection heater as a source of high energy air. Hemispherical models made of several plastic materials have been ablated and the final configuration compared with the predictions of a transient theory, which uses an integral technique. Good agreement has been found only for those plastics which sublime. For the materials which melt and

WADC TR 53-373 Sup 9

171

WADD TN 60-273 (Continued)

flow without significant vaporization, the transient theory predicts values of the surface recession which are much lower than those obtained experimentally. This discrepancy may be attributable to unrealistic values of the physical properties available for these latter materials.

The models were exposed to both a laminar and transitional heat transfer environment. The appropriate heat transfer theories required for the calculation are reviewed and discussed in detail. Corresponding heating rates have been measured experimentally and are compared with these analyses. Theory and experiment are shown to be in good agreement.

WADD TR 60-359, OTS Release

October 1960

SUBJECT: EXPERIMENTAL STUDIES TO DETERMINE THE
CHEMICAL SPECIES PREVALENT IN THE PLASMA
OF AN AIR ARC AND THE BOUNDARY LAYERS
ADJACENT TO ABLATING MATERIALS

INVESTIGATOR: R. Carrigan, E. Raisen, K. S. Schmude

CONTRACT: AF33(616)-6686, Armour Research Foundation

ABSTRACT: The objective of this work is to obtain
an increased understanding of plasma jet processes as re-
lated to the use of the plasma jet arc to test ablating
systems. Plasma jets were constructed and operated as
sources for optical and time-of-flight mass spectrometry.
Optical spectroscopic methods were used to identify
chemical species and state of excitation when inert gas or
air was used as the plasma arc working medium. Time-of-
flight mass spectrometry was not developed to the point
where species could be identified.

WADD TR 60-370, OTS Release

June 1961

SUBJECT: THERMAL RADIATION PROPERTIES OF MATERIALS

INVESTIGATOR: R. A. Seban, R. E. Rolling

CONTRACT: AF33(616)-6630, University of California

ABSTRACT: Methods are described for the measurement
of total normal emittance, in air, for temperatures up to
2500°F; for normal spectral reflectance, in air, at low
temperature; for wavelengths from 0.20 to 25 microns and in
air, at 1000°F, from 1 to 25 microns. Results of this type
are given for twenty samples of different materials and the
measured total emittances are generally within 5% of values

WADD TR 60-370 (Continued)

predicted from reflectance measurements. Reflectances were also measured as a function of angle for wavelengths of the order of 1 micron, to give absorptances as a function of angle of incidence that are useful in the appraisal of solar irradiation. A spectral emittance unit is described and the preliminary results from it for samples at 1400°F show general agreement with the measured values of spectral reflectance.

WADD TR 60-371, OTS Release

July 1961

SUBJECT: A SOURCE AND DETECTOR OF RADIATION IN
THE WAVELENGTH REGION 1500-50 ANGSTROMS
SUITABLE FOR RADIATION EFFECTS STUDIES
ON MATERIALS IN VACUO

INVESTIGATOR: H. R. Moore

CONTRACT: AF33(616)-6488, Electro-Optical Systems

ABSTRACT: This report describes a source and de-
tector of vacuum ultraviolet radiation in the wavelength
range 1500-50 angstroms which are being developed for
radiation effects studies on materials in vacuo.

The principle of the radiation source is the repetitive pulsed discharge of capacitor stored energy into a ceramic discharge tube. A very hot plasma will thus be generated which will omit vacuum ultraviolet radiation by Bremsstrahlung-like processes.

The principle of the radiation detector is photoelectric emission from a photocathode exposed directly to the radiation in vacuo. The pulsed output current from the multiplier phototube will be measured with a finite integrator.

The radiation source and detector are designed to be finally incorporated in an environmental test chamber capable of being evacuated to a working pressure of 10^{-9} mm Hg.

WADD TR 60-414, Part I, OTS Release

December 1960

SUBJECT: DEVELOPMENT OF TEST FACILITIES FOR
STUDIES IN HYPERSONIC RANGE

INVESTIGATOR: H. Halle, C. F. Price

WADC TR 53-373 Sup 9

173

WADD TR 60-414, Part I (Continued)

CONTRACT: AF33(616)-6363, University of Chicago
ABSTRACT: Hypersonic missiles re-entering the atmosphere are subjected to severe aerodynamic heating. In order to closely simulate these severe thermal conditions for testing laboratory models, a program was undertaken which consisted of the design, construction, and testing of (1) an air-stabilized a-c arc and (2) a vacuum and cooling system to evacuate the high-temperature plasma generated by the arc. Stable and reliable operation was achieved with the a-c powered arc, but it was relatively inefficient in utilizing the available power. The vacuum and cooling system to receive, cool, and exhaust to the atmosphere the plasma generated by a 1.5-megawatt air-stabilized d-c arc makes testing possible with a low-density plasma and subatmospheric stagnation pressure and permits the heat flux to be adjusted independently of the enthalpy difference.

WADD TR 60-414, Part II, OTS Release

December 1960

SUBJECT: DEVELOPMENT OF TEST FACILITIES FOR STUDIES IN HYPERSONIC RANGE
INVESTIGATOR: H. Halle, C. E. Price
CONTRACT: AF33(616)-6363, University of Chicago
ABSTRACT: Hypersonic missiles re-entering the atmosphere are subjected to severe aerodynamic heating. In order to closely simulate these severe thermal conditions for testing laboratory models, a program was undertaken which consisted of the design, construction and testing of (1) an air-stabilized a-c arc and (2) a vacuum and cooling system to evacuate the high-temperature plasma generated by the arc. The development and results of tests of three different arc configurations are presented. A double-ring electrode arc unit, when discharging into the atmosphere, provided 40 percent utilization of the available electrical power.

This a-c arc head was subsequently operated in conjunction with the previously constructed vacuum system to establish subatmospheric test conditions in the plasma stream. It was found that this arc configuration-vacuum system combination resulted in reduced thermal efficiencies.

WADD TR 60-434

November 1961

SUBJECT: THEORETICAL ANALYSIS OF THE DOWN-STREAM
INFLUENCE OF STAGNATION POINT MASS TRANSFER
INVESTIGATOR: R. J. Cresci
CONTRACT: AF33(616)-7661, Polytechnic Institute of
Brooklyn

ABSTRACT: The integral method has been applied to determine the downstream influence of homogeneous mass transfer in the stagnation region of a blunt, axisymmetric body under hypersonic flight conditions. Exponential profiles are employed in an attempt to eliminate singularities which have appeared in previous analyses utilizing polynomial profiles. The analysis is performed by introducing an additional differential equation, obtained by a second integration of the momentum equation. When used in conjunction with the Karman integral relations for the momentum and energy equations, a system of first order differential equations result which are solved by numerical means on a digital computer. The results indicate that the singularities occurring in the application of the integral method using polynomial profiles do not arise in the present analysis utilizing exponentials. The heat transfer rates obtained are compared to experimental data and are found to predict the down-stream effect of the mass injection reasonably well if the results are normalized with respect to the zero mass transfer prediction.

WADD TR 60-581, Part II

July 1961

SUBJECT: THERMAL PROPERTIES OF REFRACTORY
MATERIALS
INVESTIGATOR: J. Cape, R. Taylor
CONTRACT: AF33(616)-6794, Atomics International
ABSTRACT: Refinements in the transient thermal property apparatus are described. With these modifications, the apparatus has been used to determine the thermal diffusivity of tungsten boride from about 1300°C to 1600°C. The measured values increase from about 0.054 to 0.058 over this temperature interval. The techniques and apparatus for measuring the specific heat or brittle conductors by pulse heating are also described. Resistivity and specific heat data for uranium silicide of several compositions are reported. The resistivity and specific heat increased with increasing silicon content. For uranium silicide containing 3.8% silicon, the resistivity increased from 56 micro-ohm-cm at 0°C to 75 micro-ohm-cm at 750°C and for uranium silicide containing 5.9% silicon, the resistivity increased from 81 micro-ohm-cm at 0°C to 111 micro-ohm-cm at 800°C. The

WADD TR 60-581, Part II (Continued)

specific heat for the 3.8% silicon material is given by $c_p = 3.16 \times 10^{-6}T + 0.0412$ cal/gm-°C from 500° to 4300°C, and for the 5.8% silicon material, $c_p = 16.1 \times 10^{-6}T + 0.0455$ cal/gm-°C from 500° to 715°C, where T is in °C. The thermal conductivity of titanium carbide was measured over the temperature region 400° to 1200°C. The steady-state radial heat flow method was used. The conductivity varies linearly from 0.088 cal/sec-cm-°C at 500°C to 0.109 cal/sec-cm-°C at 1100°C. These results are in marked contrast to values reported in the literature.

WADD TR 60-657, OTS Release

January 1961

SUBJECT: THE THERMAL PROPERTIES OF SOME PLASTIC PANELS
INVESTIGATOR: P. T. Howse, Jr., C. D. Pears, S. Oglesby, Jr.
CONTRACT: AF33(616)-6073, Southern Research Inst.
ABSTRACT: The thermal expansion, specific heat, and thermal conductivity were measured for twelve different resin-reinforcement combinations, some of which had variations in reinforcement orientations. These properties were also determined for one foam core. The temperature range was generally from -50°F to 700°F. Rather extensive physical property data is also presented on all of the materials (including flexure strength, flexural modulus, Barcol hardness, density, and resin content). Detailed descriptions of the panels and cures are given.

WADD TR 60-892, OTS Release

January 1962

SUBJECT: THE DOWNSTREAM OF MASS TRANSFER AT THE NOSE OF A SLENDER CONE
INVESTIGATOR: R. Cresci, P. A. Libby
CONTRACT: AF33(616)-5944, Polytechnic Institute of Brooklyn
ABSTRACT: The influence of localized mass transfer at the nose of a slender cone under hypersonic flow conditions has been studied by experimental and theoretical means. Two gaseous coolants, nitrogen and helium are injected through a porous plug subtending a half angle of 30°. The effect of the mass transfer on the shock shape, pressure distribution, heat transfer and transition are investigated. The experimental work involved tests in the Mach number 8.0 tunnel at PIBAL. The theoretical analysis involved a study

WADD TR 60-892 (Continued)

of the effect of mass transfer on the shock stand-off distance and leads to an inviscid flow parameter permitting the experimentally determined shock shape and pressure distribution to be extrapolated to other than test conditions and to other coolant gases. Significant reductions in heat transfer are obtained with injection.

WADD TR 60-899, Part I, OTS Release

December 1960

SUBJECT: SILICA MICROBUBBLES
INVESTIGATOR: J. W. Lefforge
CONTRACT: AF33(616)-5840, Emerson & Cuming, Inc.
ABSTRACT: Glass microbubbles 0.150 to 0.020 mm diameter, bulk density 0.25g/cc, true density 0.5 to 0.8g/cc of essentially pure silica have been made by acid leaching of sodium borosilicate glass bubbles and also by fusion at 1700°C, inflation and cooling of a pure silica feed. Marked decrease in hygroscopicity, enhanced electrical properties and great increase in maximum use temperature in comparison with the commercially available 18% alkali Eccospheres resulted. Leaching resulted in reduction of alkali content from 18 to 6% with increase in use temperature from 500 to 800°C. The high temperature process, demonstrated briefly in a two pound-per-hour pilot plant scale, gave a product with essentially the properties of vitreous silica-negligible moisture absorption safe use temperature of 1300°C dielectric constant less than 1.3 and loss tangent less than 0.002. Bonding the products into structural shapes is described.

WADD TR 60-904

July 1961

SUBJECT: MEASUREMENT OF THERMAL PROPERTIES
INVESTIGATOR: I. B. Fieldhouse, J. I. Lang
CONTRACT: AF33(616)-6324, Armour Research Foundation
ABSTRACT: The purpose of this investigation has been to measure the thermal conductivity, specific heat, and linear coefficient of thermal expansion of the following materials: 1. Stainless Steel type 420, 2. Stainless Steel type 17-4PH, 3. AM 355, 4. Crucible HNM, 5. Titanium C110M, 6. Inco 713C, 7. Haynes Stellite HE 1049, 8. Kennametal K161B, 9. M252 (GE-J1500) (Solutioned 1950°F air cooled), 10. Rene 41 (GE-J1610) (Solutioned 1975°F, water quenched), 11. Vanadium Hot rolled, annealed, 12. Zirconium, 13. Molybdenum Disilicide (Slip Cast), 14. Magnesium Oxide, 15. Hafnium.

WADC TR 53-373 Sup 9

177

WADD TR 60-924, OTS Release

February 1962

SUBJECT: THE THERMAL PROPERTIES OF THIRTEEN SOLID MATERIALS TO 5000°F OR THEIR DESTRUCTION TEMPERATURES

INVESTIGATOR: D. S. Neel, C. D. Pears, S. Oglesby, Jr.

CONTRACT: AF33(616)-6312, Southern Research Inst.

ABSTRACT: The heat capacity, thermal expansion, and thermal conductivity were measured for thirteen different refractory materials, including ATJ graphite, tungsten, four nitrides, two borides, a silicate, and four carbides. The temperature range was 500° to 5000°F.

WADD TR 60-928

June 1961

SUBJECT: INVESTIGATION OF RATE AND MECHANISM OF RECOMBINATION OF MOLECULAR FRAGMENTS ON SURFACES

CONTRACT: AF33(616)-6976, Laboratories for Applied Sciences, University of Chicago

ABSTRACT: Experiments are being undertaken to study the reactions that occur when free radicals, atoms, or ions impinge upon various types of surface. A molecular beam containing free radicals, etc., is to impinge upon a prepared surface and the reaction products that are reflected or evaporated from the test surface are to be analyzed by means of an analytical mass spectrometer. Conditions will be such that the analysis is uncomplicated by reactions in the gas phase or at walls of the apparatus.

This report describes the instrumentation that has been designed and constructed for the experiments described above.

WADD TR 61-38

April 1961

SUBJECT: RADIATION CHEMISTRY OF PENTANE ISOMERS

INVESTIGATOR: R. E. Rondeau, D. R. Johnson

ABSTRACT: Three isomeric pentane gases were exposed to Cobalt 60 gamma rays and the hundred electron volt yields of the lower molecular weight products were determined. The isomers studied were normal, iso-, and neopentane. The products formed from each isomer were: hydrogen, methane, acetylene, ethane, propene, propane, butenes, and butanes. Some of the radiolysis products are explained on the basis of free radical reactions and mechanisms.

November 1961

SUBJECT: TOTAL NORMAL AND TOTAL HEMISPHERICAL
EMITTANCE OF POLISHED METALS

INVESTIGATOR: G. L. Abbott, N. J. Alvares, W. J.
Parker

CONTRACT: MIPR33(616)-60-24, U. S. Naval Radio-
logical Defense Laboratory

ABSTRACT: The objective of this research is to de-
termine the ratio of the total hemispherical to total normal
emittance for various classes of surfaces in order to find
correlation factors between the total normal emittance
usually measured and the total hemispherical emittance which
is of importance in heat transfer problems. Measurements
were made on polished platinum between 800°K and 1500°K.
The total hemispherical emittance was obtained from the
electrical power dissipation in an electrically heated strip
suspended in a vacuum of better than 10^{-4} mm Hg and its
absolute temperature measured by a thermocouple. The total
normal emittance was determined with a radiation thermopile.
The ratio of total hemispherical to total normal emittance
was also calculated directly from the angular distribution
of radiation obtained by revolving the detector about an
axis through the ribbon.

The total emittance of polished platinum
was given to within $\pm 5\%$ by the following relationships:

$$\epsilon_N = 1.03 \times 10^{-4} T(^{\circ}\text{K})$$

$$\epsilon_H = 1.22 \times 10^{-4} T(^{\circ}\text{K})$$

where the coefficients have units of deg^{-1} and hence

$$\frac{\epsilon_H}{\epsilon_N} = 1.18$$

In addition, data were taken on the spectral emittance
at 0.65 microns and on the electrical resistivity. Obser-
vations were also made on the variation of the various
measured quantities with time.

January 1960

SUBJECT: A FLASH METHOD FOR DETERMINING THERMAL
DIFFUSIVITY OVER A WIDE TEMPERATURE
RANGE

WADD TR 61-95 (Continued)

INVESTIGATOR: R. J. Jenkins, W. J. Parker
CONTRACT: MIPR33(616)-60-24, U. S. Naval Radiological Defense Laboratory
ABSTRACT: A flash method of measuring the thermal diffusivity, heat capacity, and thermal conductivity is described for the first time. A high intensity short duration light pulse is absorbed in the front surface of a thermally insulated specimen a few millimeters thick coated with camphor black, and the resulting temperature history of the rear surface is measured by a thermocouple and recorded with an oscilloscope and camera. The thermal diffusivity is determined by the shape of the temperature versus time curve at the rear surface; the heat capacity by the maximum temperature indicated by the thermocouple; and the thermal conductivity by the product of the heat capacity, thermal diffusivity, and the density. These three thermal properties are determined for copper, silver, iron, nickel, aluminum, tin, zinc, and some alloys at 20°C and 135°C and compared with previously reported values. Thermal diffusivity data are also reported for twelve different types of stainless steel over the temperature range from 20°C to 1000°C.

ASD TR 61-109

May 1961

SUBJECT: ORGANIC PERCHLORATE ESTERS
INVESTIGATOR: J. Radell, J. W. Connolly
ABSTRACT: The previously unreported n-amyl, n-hexyl, n-heptyl, and n-octyl perchlorates were prepared from the corresponding alkyl iodide and silver perchlorate. The pure perchlorate esters were stabilized as the endocycle of a urea inclusion compound. The infrared spectra and some physical properties are reported for the n-alkyl perchlorates.

ASD TR 61-110, OTS Release

May 1961

SUBJECT: EFFECT OF FLUORINE ON THE CARBONYL STRETCHING FREQUENCY OF ESTERS
INVESTIGATOR: J. Radell, L. A. Harrah
ABSTRACT: The carbonyl stretching frequencies of 19 esters of perfluorinated normal acids and 17 esters of partially fluorinated normal alcohols were measured with a precision of 0.5 cm⁻¹. The carbonyl stretching frequencies of the fluorinated esters were found to be consistently higher than the carbonyl stretching frequencies of the corresponding unfluorinated esters and dependent on the amount and position of the fluorine substituents.

ASD TR 61-110 (Continued)

The behavior of the carbonyl stretching frequency with fluorine substitution in the alcohol moiety suggests the possibility of intramolecular hydrogen bond formation between the carbonyl oxygen and the hydrogens in the alkyl chain. The presence of fluorine in this chain reduces the oxygen-hydrogen bonding in favor of a fluorine-hydrogen bond.

ASD TR 61-158, OTS Release

September 1961

SUBJECT: PERCHLORATE ESTER: PREPARATION AND POLYMERIZATION
INVESTIGATOR: J. Radell, J. W. Connolly
ABSTRACT: Epiperchloratohydrin (I) was synthesized and polymerized; although covalent perchlorates are known to be hydrolytically unstable.

Compound (I), dissolved in benzene, could be rapidly washed with water without causing major hydrolysis. A simple method for dehydrating silver perchlorate by an azeotropic distillation was used. Compound (I) before and after polymerization was explosive and required special care.

ASD TR 61-170

August 1961

SUBJECT: DETERMINATION OF IRRADIATION PRODUCED TRIPLET EXCITATION BY FLASH SPECTROSCOPY
INVESTIGATOR: J. D. McCollum, W. A. Wilson
CONTRACT: AF33(616)-7089, American Oil Company
ABSTRACT: Triplet excited states accompanying pulsed electron irradiation of solutions of anthracene, phenanthrene, naphthalene and 9-acetylanthracene have been detected by flash absorption spectroscopy. $G(\text{triplet})$ for anthracene is 0.5 for a dose of 10^{18} ev/g and a dose rate of 10^{23} ev/g-sec. Energy transfer from the solvent and subexcitation electron impact appear to be important in triplet formation. Decay rates for the above triplets are of the order 10^3 sec^{-1} in paraffin oil and 10^4 sec^{-1} in cyclohexane and benzene. The first order rate constants increase with dose and temperature. The triplet decay process is interpreted as diffusive free radical quenching by radicals produced simultaneously with triplets during irradiation. The temperature coefficient is due primarily to decrease in solvent viscosity with increasing temperature thereby increasing diffusion.

WADC TR 53-373 Sup 9

181

January 1962

SUBJECT: PREPARATION OF HIGH-PURITY SINGLE-CRYSTAL BORON

INVESTIGATOR: C. P. Talley, G. R. Taylor, Jr.

CONTRACT: AF33(616)-7498, Texaco Experiment, Inc.

ABSTRACT: Polycrystalline boron rods produced by chemical vapor plating on incandescent tungsten filaments were floating-zone melted by electron bombardment at pressures less than 10^{-5} mm Hg. During early zone-refining trials, a single crystal of β -rhombohedral boron approximately 1.5 mm in diameter and over 1 cm in length was produced.

ASD TR 61-491

November 1961

SUBJECT: RESEARCH ON ORGANIC RADIATION CHEMISTRY

INVESTIGATOR: G. A. Swan

CONTRACT: AF61(052)-283, King's College, University of Durham

ABSTRACT: Part I discusses the isomeric-ratio of 2-, 3- and 4-bromobiphenyl, formed by γ -radiolysis of bromobenzene. Part II discusses the γ -radiolysis products of trimethylamine, N-methylpiperidine and N-methyl-diethylamine.

ANALYTICAL TECHNIQUES

WADC TR 59-107, Part III, OTS Release

August 1961

SUBJECT: A MASS SPECTROMETER SYSTEM FOR MATERIALS RESEARCH (PHASE III)
INVESTIGATOR: C. F. Robinson, N. W. Bell, G. D. Perkins, R. H. Small
CONTRACT: AF33(616)-5571, Bell & Howell Research Center
ABSTRACT: There are three areas in which mass spectrometry may be expected to contribute importantly to science and technology within the foreseeable future. They are (1) identification and estimation of minor components and trace impurities in solids, (2) structural studies of high-molecular weight materials, (3) identification and estimation of unknown materials by precise measurement of molecular weight. At the start of this program, no mass spectrometer had been built which was capable of use in all three of these fields. A three-phase program of research and development was undertaken to produce a single high-performance mass-resolving system which, together with a limited number of ion source and ion detector modules, would be adaptable to any of the three problem areas. A basic system with a resolution of about one part in 2,500 has been produced. Ion sources using spark, electron bombardment and high temperature crucible have been adapted to the basic system and photographic plate, electrometer and electron multiplier detectors have been used. A complete instrument embodying all of these features is now at the Materials Central.

WADD TR 60-482, OTS Release

March 1961

SUBJECT: ANALYSIS OF GASES IN METALS
INVESTIGATOR: J. J. Schmidt-Collerus, A. J. Frank
CONTRACT: AF33(616)-6381, Denver Research Inst.
ABSTRACT: A study has been made to investigate the feasibility of fusion extraction of gases from metals in combination with gas-solid adsorption chromatography for the analysis of gases in metals. The resolution of extracted nitrogen, hydrogen, oxygen and carbon monoxide by a molecular sieve 5A chromatographic column is demonstrated. An apparatus which combines gas extraction from metallic samples, gas transfer and chromatographic analysis is described and recommendations presented for further improvement of the system operation. The application of the basic

WADD TR 60-482 (Continued)

principle for the determination of nitrogen in magnesium and ingot iron has been investigated. While the feasibility of the basic principle could be demonstrated, failures in apparatus shortcomings prevented sufficient quantitative information; corrective measures are proposed. Extension of the analytical method to inert-gas fusion extraction is discussed.

WADD TR 60-655, OTS Release

March 1961

SUBJECT: THERMODYNAMICS OF THE INTERACTION OF NIOBIUM AND TANTALUM WITH OXYGEN AND NITROGEN AT TEMPERATURES NEAR THE MELTING POINT

INVESTIGATOR: J. Paul Pemsler

CONTRACT: AF33(616)-6627, Nuclear Metals, Inc.

ABSTRACT: The concentration of nitrogen and oxygen in niobium and tantalum in equilibrium with the pure gas has been determined as a function of pressure at three temperatures near the melting point, and in the liquid phase at the melting point. The solubility of nitrogen in niobium is directed proportional to the square root of the pressure (Sievert's Law) up to the solubility limit, where the saturated solution is in equilibrium with Nb_2N . The solubility of nitrogen in tantalum shows a negative deviation from Sievert's Law at nitrogen concentrations above 5 atomic percent; beyond the solubility limit saturated solution is converted to Ta_2N . The niobium-oxygen and tantalum-oxygen systems obey Sievert's Law up to the solidus point. Temperatures investigated were above the melting points of metal oxides and no oxide scales were obtained. A solubility inversion was noted in the tantalum-oxygen system where the oxygen concentration in equilibrium with a specified pressure of oxygen is lower at 2850 than at 2960°C. Data was used to calculate the partial molar and integral values of the free energy, enthalpy, and entropy of dissociation nitrogen in niobium and tantalum.

WADD TR 60-759, OTS Release

December 1960

SUBJECT: INFRARED SPECTRA OF THE PHENYL COMPOUNDS OF GROUP, IVb, Vb, and VIb ELEMENTS

INVESTIGATOR: L. A. Harrah, M. T. Ryan, C. Tamborski

ABSTRACT: Infrared spectra between 2 and 35 microns of a large number of phenyl compounds of group IVb, Vb, and

WADC TR 53-373 Sup 9

184

WADD TR 60-759 (Continued)

VIIb elements are presented. The spectral features of interest in qualitative and quantitative analysis are discussed and empirical correlations between the spectra and properties of the central metal atom demonstrated. The spectra beyond 15 microns are shown to be particularly useful in the quantitative identification of this class of materials.

ASD TN 61-78

September 1961

SUBJECT: DETERMINATION OF TRACE IMPURITIES IN
HIGH PURITY TUNGSTEN METAL
INVESTIGATOR: K. F. Sugawara
ABSTRACT: This report includes results from a literature survey initiated to determine the feasibility of analyzing high purity tungsten metal for metallic trace impurities to ascertain whether its significance as a refractory metal in the current research on high temperature effects warrants its inclusion as a "systems" project as a project task for internal development. This report includes findings on effects of impurities on metallurgical properties, and different methods for effecting separation and concentration of impurities. Additionally, based on the literature search, the author proposes a "best" method for analysis.

ASD TN 61-135, OTS Release

December 1961

SUBJECT: RAPID MEANS OF MONITORING THE THERMAL
DECOMPOSITION OF MAGNESIUM PERCHLORATE -
CHLORIDE DETERMINATION
INVESTIGATOR: K. F. Sugawara
ABSTRACT: A rapid procedure by which the thermal decomposition of magnesium perchlorate can be monitored, is discussed. The method, based upon the spectrophotometric measurement of the iron (III) chloride complex, is quite simple and accurate.

ASD TR 61-169

June 1961

SUBJECT: DOSE RATE EFFECTS IN LIQUID HYDROCARBON
RADIOLYSIS
INVESTIGATOR: T. D. Nevitt, W. A. Wilson
CONTRACT: AF33(616)-7089, American Oil Co.
ABSTRACT: Previous hydrocarbon radiolyses have

ASD TR 61-169 (Continued)

shown product differences that might have been attributed to dose rate differences. During this study, the effects of extremely high dose rates on products of hydrocarbon radiolyses were examined. Cyclohexane, hexane, and pentane were irradiated at 2×10^{16} , 5×10^{23} ev/g-sec. Radiation dose ranged from 10^{19} to 10^{20} ev/g. The products from cyclohexane: hydrogen, cyclohexene and dicyclohexyl did not change with dose rate. Experiments using cyclohexene for hydrogen scavenging or deuterium labeling gave no evidence for dose rate effects occurring in radiation spurs. Low molecular weight products from hexane change slightly with dose rate; C_{12} products from hexane and C_{10} products from pentane show small but definite dose rate dependence. At the high dose rates investigated, dose rate does not control product ratios. The results indicate a more marked dependence on dose rate might be found at doses lower than examined here.

ASD TR 61-176

June 1961

SUBJECT: THE DEVELOPMENT OF A HIGH TEMPERATURE (1000°C) GAS CHROMATOGRAPH
INVESTIGATOR: C. Euston, A. Martin
CONTRACT: AF33(616)-7160, F&M Scientific Corp.
ABSTRACT: A gas chromatograph operable at temperatures up to 1000°C is described. Featuring either isothermal or linear programmed temperature operation, the unit utilizes a novel integrated column and hydrogen flame ionization detector for sensitivity and stability. The stainless steel column is heated by passing high currents (up to 120 amperes) directly through the metal. Other wrapped heater columns may also be used. Design considerations, descriptive information and performance data for the instrument are presented. Organic analysis present no problem, and the detection of organo-metallic compounds appear straight forward. Application to inorganic chloride salts has given anomalous results. The data indicate that reaction of the sample is the primary cause of this behavior, and that proper choice of column materials and sampling techniques will be required for these analyses.

ASD TR 61-183

June 1961

SUBJECT: ANALYSIS OF REFRACTORY METALS USING A NEW SOLID SOURCE DOUBLE FOCUSING MASS SPECTROMETER

ASD TR 61-183 (Continued)

INVESTIGATOR: W. Baun, D. Fischer
ABSTRACT: A newly developed sample-spark source mass spectrometer capable of sensitivities of 0.1 ppm to 0.01 ppm is briefly described. Analytical techniques, Methods, and results are presented for the analysis of refractory metals. Emphasis is placed on trace elements analysis of high purity single crystal and polycrystalline tungsten and comparisons are made concerning methods of purification. Data are also shown for analysis of tungsten in which a dispersed second phase such as thoria exists. Analysis of other components purposely introduced to tungsten to increase yield and creep rupture strength is discussed.

ASD TR 61-202

September 1961

SUBJECT: A STUDY OF THE INFRARED SPECTRA OF SOME BRANCHED CHAINED ALIPHATIC BROMIDES AND IODIDES
INVESTIGATOR: N. T. McDevitt
ABSTRACT: The fundamental vibration of the carbon halide stretching frequency in aliphatic compounds may occur at more than one frequency for the same compound. These so called extra frequencies are due to the different rotational isomers of the compound. Identification of the various branched chained aliphatic mono halides can be obtained from the study of the frequencies of their individual rotational isomers.

ASD TR 61-407, OTS Release

November 1961

SUBJECT: RESEARCH ON POLYMERIC MATERIALS SUITABLE FOR USE AS BINDERS FOR SOLID PROPELLANTS
INVESTIGATOR: S. J. Chlystek
CONTRACT: AF33(616)-8127, Armstrong Cork Co.
ABSTRACT: Several polymeric binders suitable for use as solid propellant binders were prepared and evaluated by a variety of physical and chemical tests. The basic chemistry involved preparation of suitable polyesters and diisocyanate-terminated polymers, which were combined in appropriate proportions and under suitable conditions to yield soft, flexible polyurethane elastomers.

The elastomeric materials were studied by continuous and intermittent stress relaxation, low temperature retraction, equilibrium swelling volume, and

ASD TR 61-407 (Continued)

dynamic and static stress-strain measurements. Energies of activation for the rupture of bonds in strain at elevated temperatures were calculated from Arrhenius plots of relaxation time versus elapsed time. The glass transition temperature was predicted from the low temperature retraction data, and other physical characteristics dependent on polymer structure were inferred from this data.

ASD TR 61-442, OTS Release

November 1961

SUBJECT: X-RAY DIFFRACTION STUDY OF STRAIGHT CHAIN CARBOXYLIC ACIDS (C₁ to C₁₄)
INVESTIGATOR: W. L. Baun
ABSTRACT: X-Ray diffraction effects in solid state saturated fatty acids are presented with emphasis on short chain fatty acids normally liquid at room temperature. Long and short spacings are determined, anisotropic thermal expansion is shown, polymorphic tendencies are investigated, and other variable temperature effects are discussed.

ASD TR 61-722, OTS Release

March 1962

SUBJECT: INVESTIGATION OF THE INFRARED ABSORPTION SPECTRA OF SELECTED AROMATIC COMPOUNDS
INVESTIGATOR: R. J. Jakobsen
CONTRACT: AF33(616)-7162, Battelle Memorial Inst.
ABSTRACT: As the first step in making assignments of the low-frequency vibrations of para-substituted benzene compounds, complete vibrational assignments of several representative compounds have been started. While assignments have been made for many monosubstituted benzenes, there is need for a complete assignment of a mono-substituted benzene with an electron-attracting substituent. To fill this need, an assignment of benzonitrile has been made. Attempts to prepare benzonitrile-d₅ were unsuccessful, but are being continued. To confirm the previous assignment of phenol, a sample of phenol-2,4,6-d₃ was prepared. A complete vibrational assignment of this molecule has been made. Attempts to prepare phenol-d₅ are in progress. The assignments of p-cresol and deuterated p-cresols has been carried almost to completion. Using the vibrational assignments mentioned above as a foundation, the low-frequency vibrations of 28 para-substituted benzene compounds have been assigned as completely as possible. It has been shown that certain of the low-frequency vibrations are sensitive to the nature of the substituent.

ASD TDR 62-32, Part II, OTS Release

February 1962

SUBJECT: TRIPLET EXCITATION INDUCED BY ELECTRON IRRADIATION. PART II. Examination of Aromatic Systems
INVESTIGATOR: J. D. McCollum, W. A. Wilson
CONTRACT: AF33(616)-8247, American Oil Co.
ABSTRACT: Flash spectroscopy has been used to search for transient triplet absorption from eleven compounds irradiated by single electron pulses. Transients ascribed to triplets were found from 1,2-benzanthracene and p-terphenyl. Quenching of triplets in viscous paraffin oil appears sensitive to triplet structure; relative quenching rates of 8:3:1 were observed for p-terphenyl: 1,2-benzanthracene: anthracene triplets. Phenyl ether gives a transient probably assignable to phenyl radical.

ASD TDR 62-179, OTS Release

February 1962

SUBJECT: ELECTROMETRIC METHODS OF ANALYSIS
INVESTIGATOR: J. F. Gettings
ABSTRACT: This report includes a discussion of instruments used in electrometric analysis and the feasibility of using the instrument for several types of analysis. A discussion of new automated instrument and new technique for electrometric analysis will be reviewed which was presented at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. A study was made to find the most rapid method for the determination of chlorine and bromine in organic compound using microanalytical techniques. The results obtained from this study enable the micro-analytical laboratory to obtain an analysis in approximately fifteen minutes.

ASD TDR 62-224, OTS Release

March 1962

SUBJECT: THE INFRARED SPECTRA AND VIBRATIONAL ASSIGNMENTS FOR SOME ORGANO-METALLIC COMPOUNDS
INVESTIGATOR: F. W. Behnke, C. Tamborski
ABSTRACT: The infrared spectra from 850 - 250 cm^{-1} and the vibrational assignments of nine organometallic compounds are reported. The compounds investigated were members of the series $(\phi)_3\text{M-A}$ where $\text{M} = \text{Si, Ge, Sn}$ and $\text{A} = \text{H, Cl, Br}$.

From the infrared spectra which were ob-

ASD TDR 62-224 (Continued)

tained it was found that the region from 800 - 250 cm^{-1} contains analytical bands for most of the nine compounds which were investigated.

ASD TDR 62-226, OTS Release

February 1962

SUBJECT: RAPID METHOD FOR THE MICRO DETERMINATION OF PHOSPHORUS IN ORGANIC COMPOUNDS
INVESTIGATOR: H. M. Rosenberg, T. M. Downer
ABSTRACT: This report contains an evaluation of a rapid volumetric procedure for the micro determination of phosphorus in organic compounds. The method is based on combusting the sample in a Schöniger flask followed by titration with cerous solution, using Eriochrome Black T as the indicator.

ASD TDR 62-227, OTS Release

February 1962

SUBJECT: A RAPID METHOD FOR THE MICRO DETERMINATION OF CARBON AND HYDROGEN IN ORGANIC COMPOUNDS
INVESTIGATOR: H. M. Rosenberg, T. M. Downer, C. R. Riber
ABSTRACT: This report describes a procedure for the rapid determination of carbon and hydrogen in organic compounds. The analysis can be accomplished in 30 minutes, using samples weighing between 3 and 5 milligrams. A new technique was developed, using a modified Pregl combustion train.

ASD TDR 62-325, OTS Release

April 1962

SUBJECT: DEVELOPMENT OF A METHOD FOR THE INSTRUMENTAL DETERMINATION OF OXYGEN IN ORGANIC COMPOUNDS CONTAINING NITROGEN, SULFUR AND HALOGENS
INVESTIGATOR: H. S. Haber, K. W. Gardiner
CONTRACT: AF33(616)-7762, Bell & Howell Research Center
ABSTRACT: This report contains the results of research efforts on the development of an instrument for the determination of chemically-bound oxygen in organic compounds containing nitrogen, sulfur, and halogens. Several different approaches to the problem are reviewed and the most

ASD TDR 62-325 (Continued)

promising approach is discussed in detail. Research prototype instruments have been constructed and selected analytical data obtained with these instruments are presented.

DESIGN CRITERIA

WADC TR 59-595, Part II, OTS Release

August 1961

SUBJECT: METALLURGICAL AND MECHANICAL CHARACTERISTICS
OF HIGH-PURITY TITANIUM-BASE ALLOYS
INVESTIGATOR: A. G. Ingram, D. N. Williams, R. A. Wood,
H. R. Ogden, R. I. Jaffee
CONTRACT: AF33(616)-5462, Battelle Memorial Inst.
ABSTRACT: The relationships between mechanical
properties, alloy composition, microstructure, and thermal
history have been studied for high-purity titanium-base
alloys. The alloy systems investigated include Ti-Al, Ti-
Zr, Ti-Hf, Ti-Al-Zr, Ti-Al-Hf, Ti-Al-Cb, Ti-Ch, Ti-W, Ti-
V-Cr, and Ti-V-Cr-Al. Mechanical-property data, including
tensile and flow properties, impact behavior, hardness,
aging, and cooling-rate data were determined. The metallurgical
principles involved are discussed.

WADC TR 59-744, Vol I

June 1961

SUBJECT: INVESTIGATION OF FEASIBILITY OF UTILIZING
AVAILABLE HEAT RESISTANT MATERIALS FOR
HYPERSONIC LEADING EDGE APPLICATIONS
INVESTIGATOR: F. M. Anthony, F. A. Merrihew, A. L.
Mistretta, W. H. Dukes
CONTRACT: AF33(616)-6034, Bell Aerosystems Co.
ABSTRACT: The purpose of this contract was to in-
vestigate the feasibility of utilizing available heat re-
sistant materials in the fabrication of leading edges for
hypersonic gliders. This particular volume summarizes the
extensive analytical studies conducted from the literature
surveys to the testing of the molybdenum alloy and graphite
leading edge assemblies produced.

By applying proper design philosophies
to account for present material limitations, it is feasible
to produce leading edges for hypersonic gliders by utilizing
available materials. In fact, the designs produced appear
to be suitable for service use for specific applications.
Thus, the study demonstrated more than feasibility, it in-
dicated the practicability of immediate utilization of
available materials.

To avoid possible security problems
which might classify this volume, performance capabilities are
not summarized. They are presented only in Volume IX.

March 1961

SUBJECT: INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOL II. Analytical Methods and Design Studies

INVESTIGATOR: F. M. Anthony, A. H. Blessing, W. H. Buckley, W. H. Dukes

CONTRACT: AF33(616)-6034, Bell Aerosystems Co.

ABSTRACT: An investigation of the feasibility of utilizing available heat resistant materials for hypersonic leading edge applications was made. Analytical methods for relating the many flight path, vehicle, component, and material parameters were established. Using the methods developed the various parameters were investigated as to their effects on maximum operating temperature and on thermal stresses. Experimental results are compared with predictions. Two leading edge designs, one of coated molybdenum and one of coated graphite were produced.

October 1960

SUBJECT: INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOL IV. Thermal Properties of Molybdenum Alloy and Graphite

INVESTIGATOR: I. B. Fieldhouse, J. I. Lang

CONTRACT: AF33(616)-6034, Bell Aircraft Corp.

ABSTRACT: The purpose of this contract was to investigate the feasibility of utilizing available heat resistant materials in the fabrication of leading edges for hypersonic boost-glide vehicles. This particular volume presents the results of measurements of the thermal conductivity, specific heat, linear thermal expansion, and emittance of a 0.5% titanium alloy molybdenum, and of siliconized ATJ graphite as a function of temperature. Emittance measurements were made on coated and uncoated materials.

July 1961

SUBJECT: INELASTIC DESIGN OF LOAD CARRYING MEMBERS Part IV. The Behavior of Beam-Columns in the Inelastic Range

INVESTIGATOR: B. B. Muvdi, O. M. Sidebottom

CONTRACT: AF33(616)-7600, University of Illinois

WADD TR 60-580, Part IV (Continued)

ABSTRACT: Two theories were presented for constructing either moment-load or load-deflection relations as well as the collapse loads for beam-columns. In each case, trial and error solutions were required which used constant depth of yielding interaction curves. A "so-called" exact theory was presented which gave results as accurate as desired; however, the theory was not practical because of the excessive time required. An approximate theory was presented which gave results in close agreement with the exact theory and with experimental data. This theory required the elastic solution for maximum elastic conditions. The experimental part of the investigation included tests of rectangular - and T-section columns made of 2024-T4 aluminum alloy, SAE 1020 steel, and 17-7PH stainless steel. Several slenderness ratios were considered. In addition to the variable axial load, the columns were subjected to a constant transverse load either at midspan or at quarter span which produced a bending stress of 0.25, 0.50, or 0.75 σ_y .

WADD TR 60-654

January 1961

SUBJECT: PROPERTIES OF PLASMA SPRAYED MATERIALS
INVESTIGATOR: M. A. Leinstein
CONTRACT: AF33(616)-6376, General Electric Co.
ABSTRACT: Various mechanical and physical properties of selected high melting point metals, metalloids and oxides deposited by the plasma deposition process were determined and compared with the properties of these materials produced by conventional processes. These materials include W, Mo, Ta, HfC, TaC, ZrB₂, CeO₂, HfO₂, Cr₂O₃, and Cr-Al₂O₃. The properties studied were microstructure, crystallographic structure, thermal expansion, enthalpy, specific heat, chemistry, strength elongation, modulus of elasticity, density, hardness and resistance to thermal shock. Properties were determined both at room temperature and at elevated temperatures. Tungsten and molybdenum were sprayed from both the wire and powder form; but Ta, the metalloids, and the oxides were in powder form only. The metals and metalloids were sprayed in air and in a controlled environment. The oxides were sprayed in air only. Spraying parameters were established for two types of plasma equipment: a 40 KW non-consumable electric powder gun; and a 12 KW consumable electric arc wire gun. Two environmental tanks were used for spraying under controlled atmospheres at near ambient and reduced pressures.

July 1961

SUBJECT: INVESTIGATION OF TUNGSTEN-TANTALUM-COLUMBIUM-BASE ALLOYS

INVESTIGATOR: C. R. McKinsey, A. Leslie Mincher,
W. F. Sheely, J. L. Wilson

CONTRACT: AF33(616)-7225, Union Carbide Metals Co.

ABSTRACT: Two categories of properties pertaining to selected refractory alloy systems are presented. First, the alloys were screened as to their usefulness as high-temperature structural materials (Parts II and III). Measurements of hot-hardness and oxidation resistance of arc-cast specimens delineated areas within the tungsten-tantalum-columbium ternary system combining high hardness at temperatures up to 2900°F, with improved oxidation resistance at 2190°F. The addition of tungsten and titanium was found to improve the oxidation resistance of tantalum at 1830 and 2190°F., probably because of the formation of complex oxides in the other scale. The second area of investigations is concerned with mechanical properties of tungsten or columbium at low and intermediate temperatures (Parts IV through VII). The low-temperature yield properties of tungsten were studied, employing a compression test. Static strain-aging of tungsten at 400 to 600°C was attributed to hydrogen. Dynamic strain-aging of columbium-oxygen alloys occurred at 300 to 400°C, while the addition of titanium, zirconium, and vanadium to columbium was found to shift the strain-aging region to 700 to 800°C.

April 1961

SUBJECT: MECHANICAL PROPERTIES OF TWO TITANIUM FORGING ALLOYS Ti 155A and C135AMo

INVESTIGATOR: R. G. Henning

ABSTRACT: Mechanical properties of two annealed titanium forging alloys, Ti 155A and C135AMo, were obtained. These properties included tensile, compressive, pin shear, bearing, and notched tensile at room, 200, 400, 600, 800, and 1000°F temperatures. Stressed and non-stressed exposure tests were conducted at 600, 800, and 1000°F. Torsion tests were conducted at room temperature only.

Properties vs. temperature graphs and tensile, compressive, and bearing stress-strain curves are presented. The ratio of the various properties to room temperature tensile results are plotted vs. temperature.

August 1961

SUBJECT: EFFECT OF CREEP-EXPOSURE ON MECHANICAL PROPERTIES OF RENE' 41

INVESTIGATOR: J. V. Gluck, J. W. Freeman

CONTRACT: AF33(616)-6462, University of Michigan

ABSTRACT: Rene' 41 was tensile tested at room temperature after creep-exposure or unstressed exposures of 10 to 200 hours at temperatures between 1200° and 1800°F. Thermally-induced structural changes reduced strength and ductility after exposures above 1500°F, while creep strains caused increased strength and decreased ductility after lower temperatures. Limited data indicated that surface reactions reduced properties in addition to the effects of the creep-exposures. Creep accelerated structural changes and raised yield strength by a Bauschinger effect. Ductility was reduced more after creep than by thermally-induced changes. At 1200° and 1300°F, creep strains approaching the ultimate ductility induced deep cracking. This effect was not found at higher temperatures. The loss in strength from thermal changes appeared to be due to the overaging of the gamma prime precipitate, while losses in ductility were associated with build-up of carbides and agglomeration of gamma prime at the grain boundaries. Surface effects were associated with intergranular oxidation, cracking, and alloy depletion. The role of creep needs further clarification and further study of surface reactions is needed before general principles can be formulated for the influence of creep-exposure on normal temperature mechanical properties.

November 1961

SUBJECT: EFFECT OF CREEP-EXPOSURE ON MECHANICAL PROPERTIES OF RENE' 41. Part II. Structural Studies, Surface Effects, and Re-Heat Treatment

INVESTIGATOR: J. V. Gluck, J. W. Freeman

CONTRACT: AF33(616)-6462, University of Michigan

ABSTRACT: The effect of creep-exposure on room temperature mechanical properties of Rene' 41 was studied for temperatures of 1200-1800°F and times up to 200 hours. Unstressed exposures were for as long as 2012 hours at 1700°F. Thermally-induced structural changes reduced strength and ductility after exposures at 1400-1800°F. Reduced yield strength was due to decreased volume fraction of gamma prime and secondarily to an increase in the particle size. Ductility was reduced by formation of massive grain boundary carbides. Up to 1500°F, creep caused strain

ASD TR 61-73, Part II (Continued)

hardening and Bauschinger effects. Except for surface effects, damage was restorable by re-heat treatment. Yield strength was restored by re-solution and reaging to produce fine gamma prime. Complete re-solution of carbides was required to restore ultimate strength and ductility. Microcracking was not observed. Creep induced intergranular surface cracking at 1200-1300°F which reduced ductility. Surface effects for exposures above 1400°F were thermally induced. General principles were formulated for damage to properties of nickel-base alloys.

ASD TN 61-117, Part II

November 1961

SUBJECT: FATIGUE OF METALS - CORROSION AND HEAT
RESISTANT METALS - Section I
CONTRACT: AF33(616)-7238, Belfour Engineering Co.
ABSTRACT: The graphs presented herein display metals
fatigue information from various sources of published and
unpublished test reports which have been processed and re-
generated through a semi-automatic data processing system.
Each series or set of graphs contain descriptive information
(legends) which identifies the material, test procedure,
test conditions and the most significant test and/or material
variables associated with the plotted data. The data dis-
played in each set of graphs is intended to answer very
general "questions" and to serve as a guide to further in-
vestigation of specific areas within the subject presented.

ASD TN 61-117, Part III

February 1962

SUBJECT: EFFECT OF ULTIMATE TENSILE STRENGTH ON
THE FATIGUE BEHAVIOR OF 4340 STEEL
INVESTIGATOR: Lt D. Ingles
CONTRACT: AF33(616)-7238, Belfour Engineering Co.
ABSTRACT: The graphs presented herein display
metals fatigue information from various sources of pub-
lished and unpublished test reports which have been
processed and regenerated through a semi-automatic data
processing system. Each series or set of graphs contain
descriptive information (legends) which identifies the
material, test procedure, test conditions and the most signi-
ficant test and/or material variables associated with the
plotted data. The data displayed in each set of graphs is
intended to answer every general "question" and to serve as
a guide to further investigation of specific areas within the
subject presented.

August 1961

SUBJECT: PROPERTIES OF YTTRIUM AND THE RARE
EARTH METALS - OXYGEN AND ALLOY SYSTEMS
INVESTIGATOR: B. Love
CONTRACT: AF33(616)-6829, Research Chemicals Div.,
Nuclear Corp. of America
ABSTRACT: Alpha yttrium and erbium are miscible.
In the yttrium-neodymium system there is partial solubility
and an intermediate phase is present. The solubility of
oxygen is low in yttrium, erbium, neodymium, and samarium.
Alpha-beta transformations are essentially unaffected. A
high temperature monoxide is proposed. The solubility of
erbium in cobalt is low. The first compound is $\text{Co}_{17}\text{Er}_2$.

Tantalum and niobium form extensive liquid immiscibility regions with rare earths, terminating in a monotectic near the tantalum (niobium) and of the systems; a eutectic at the rare earth end. Solubility is very low. Improved atmospheric corrosion resistance was found for some niobium and cobalt compositions with rare earth additions. The tensile properties of yttrium were improved by alloying with erbium or zirconium. Purification of yttrium and erbium, and improved analytic methods are described. Beryllium with erbium additions shows grain refinement.

ASD TR 61-145

SUBJECT: WEATHERING OF GLASS-FABRIC-BASE PLASTIC
LAMINATES UNDER STRESS
INVESTIGATOR: K. E. Kimball
CONTRACT: AF33(616)-58-1 & 33(616)61-06, Forest
Products Laboratory
ABSTRACT: To determine the effect of weathering
on the mechanical properties of a reinforced plastic panel
under stress, three laminates were exposed in the stressed
and non-stressed condition for periods up to 3 years. After
3, 12, and 36 months, the tensile, compressive, and flexural
strength properties were determined. Weathering generally
had about the same effect on strength properties of stressed
panels as on non-stressed ones. Outdoor weathering in
Florida resulted in a greater reduction in strength properties
of the polyester laminate than those of the epoxy or phenolic
laminates.

August 1961

SUBJECT: THEORETICAL FORMABILITY. VOL II. -
Application
INVESTIGATOR: W. W. Wood
CONTRACT: AF33(616)-6951, Chance Vought Corp.
ABSTRACT: This two-volume report presents methods of determining formability analytically for the twelve most common processes of forming sheet metal. This method is based on utilization of a material's mechanical properties to predict formability. This volume on application is presented in handbook form giving design and manufacturing information for the nineteen materials in the program. These materials covered some of the most currently used alloys in the following categories: (1) magnesium, (2) aluminum, (3) titanium, (4) stainless steel, (5) tool steel, (6) nickel and cobalt base, and (7) the refractory metals. Graphs, equations, and design tables are presented for each process.

ASD TR 61-216, OTS Release

SUBJECT: PRESENTATION OF CREEP DATA FOR DESIGN PURPOSES
INVESTIGATOR: M. N. Arnes, M. M. Tuttle
CONTRACT: AF33(616)-7201, Boeing Airplane Co.
ABSTRACT: This program was conducted to obtain additional and comparative creep data, to compare creep data from several sources, and to recommend what values to present and in what form to present creep data for design purposes.

Conventional long time creep tests were performed on A-286 at 1200°F and 1500°F; Al10AT at 800°F and 1000°F; and Unimach 2 at 600°F and 900°F for purposes of comparing with existing similar data. Conventional creep tests were performed on Rene' 41 at 1250, 1400, 1550, 1700, 1850, and 2000°F.

Data were analyzed and are presented in the form of activation series equations. Nomographs were derived for each material.

Cyclic creep tests were performed on Rene' 41 in which both stress and temperature were cycled. Cyclic data were found to be comparable to the constant stress constant temperature data.

September 1961

SUBJECT: MECHANICAL PROPERTIES OF SOLUTION-TREATED
TITANIUM SHEET ALLOY B120VCA

INVESTIGATOR: R. G. Henning

ABSTRACT: Mechanical properties of three heats of solution-treated titanium sheet alloy B120VCA were obtained. These properties included tensile compressive, sheet single shear, bearing and 105° bend at temperatures of 200°, 400°, 600°, 800°, and 1000°F. All properties were determined in both the transverse and longitudinal rolling directions.

Curves are presented for mechanical properties vs. test temperature and the ratio of room temperature tensile properties to compressive, bearing, and shear properties vs. temperature. Typical stress-strain curves for tensile and compression tests were drawn for all test temperatures.

September 1961

SUBJECT: EFFECT OF CREEP-EXPOSURE ON MECHANICAL
PROPERTIES OF 80Ni - 20Cr AND TZM
MOLYBDENUM

INVESTIGATOR: J. V. Gluck, J. W. Freeman

CONTRACT: AF33(616)-6462, University of Michigan

ABSTRACT: The effect of creep-exposure at 1000° to 1800° on room temperature mechanical properties was studied for a relatively structurally stable 80Ni-20 Cr alloy. Strain hardening and residual stress raised strength and lowered ductility after creep at the lower temperatures. Internal grain boundary micro-cracking increased with temperature and creep strain. Very large amounts of cracking were required to significantly reduce strength and ductility. For a given exposure, fine grain material was less damaged by cracking than coarse grain material. The fine grain material was also subject to loss in strength from grain growth. The low strength and high ductility of the alloy probably reduced the damage from cracking. Impurities and minor phases may have affected results. A higher strength material would have shown more damage due to the added effects of increased notch sensitivity. No conclusive results were obtained with the TZM molybdenum alloy due to experimental difficulties and uncertainties of the material.

March 1962

SUBJECT: DEVELOPMENT OF A Ti-Al-Cb ALLOY FOR USE
AT 1200° - 1800°F
INVESTIGATOR: J. B. McAndrew, C. R. Simcoe
CONTRACT: AF33(616)-7262, Armour Research Foundation
ABSTRACT: Titanium-base alloys containing major
amounts of columbium and aluminum are being studied with the
object of developing new high-temperature alloys of low den-
sity. This report presents the findings of the second year
of this program, derived from the preparation and examina-
tion of: 35 quaternary alloys containing small additions of
tin, hafnium, or zirconium; 4 high-purity ternary alloys;
and ten-pound melts of Ti-15Cb-10Al and Ti-17.5Cb-15Al.

In a number of alloys, improved tensile properties and oxidation resistance resulted from the addition of tin, hafnium, or zirconium, and in some instances very high strength-density ratios were maintained up to 1800°F. The properties of high-purity alloys were similar to those of alloys prepared with sponge titanium. It is recommended that further effort should be directed toward the more highly alloyed compositions, including those containing hafnium and zirconium.

January 1962

SUBJECT: INVESTIGATION OF THE EFFECTS OF RAPID
LOADING AND ELEVATED TEMPERATURES ON THE
MECHANICAL PROPERTIES OF COMPRESSIVE
AND COLUMN MEMBERS
INVESTIGATOR: P. R. Dioguardo, R. D. Lloyd
CONTRACT: AF33(616)-7345, Marquardt Corp.
ABSTRACT: The short time tensile and compressive
properties were evaluated from Rene' 41 and Haynes 25 sheet
alloys at room temperature and 800° to 2200°F. Tests con-
ducted at various strain rates (0.00001 to 0.1 in./in./sec)
produced significant differences in strength, particularly
at elevated temperatures.

Creep properties, both tensile and com-
pressive, were determined for the same alloys at tempera-
tures of 1600° to 2200°F and times up to 15 minutes. At
the higher stress levels, creep rates became very rapid and
considerable amounts of plastic strain occurred in times of
1 second or less.

A preliminary study was conducted on the
use of programmed, forced strain rate tests to predict short
time creep properties.

ASD TR 61-497

April 1962

SUBJECT: SHEAR STRENGTH OF ADHESIVES IN STAINLESS
STEEL AND ALUMINUM LAP JOINTS AT
TEMPERATURES FROM -100° to 1000°F
INVESTIGATOR: W. Z. Olson, R. M. Lulling
CONTRACT: DO(616)-61-06, Forest Products Laboratory
ABSTRACT: Data are given on the effect of various
temperatures and methods of heating on specimens bonded with
various commercial adhesive systems. Shear strength was re-
duced more by rapid infrared heating than by oven heating to
the same temperature levels.

ASD TR 61-516

September 1961

SUBJECT: STATISTICAL EVALUATION OF RENE' 41
TENSILE PROPERTIES
INVESTIGATOR: D. P. Moon, G. H. Beatty, R. J. Favor
CONTRACT: AF33(616)-6410, Battelle Memorial Inst.
ABSTRACT: Results of routine acceptance tests at
room temperature and at 1400°F show that the variation in
tensile strength, yield strength, and ductility among heat-
treated Rene' 41 sheet specimens from a given sheet often
exceeds that resulting from the combined effects attributable
to producer, thickness, and heat. The distribution of
strength properties is not normal in the tails; in part-
icular, the frequency of values is higher than normal in the
low-strength tail, from which design values are usually
selected. This fact must be taken into account in selecting
design values based on probability of failure.

ASD TR 61-684, OTS Release

March 1962

SUBJECT: SCALE-UP DEVELOPMENT OF TANTALUM-BASE
ALLOYS
INVESTIGATOR: H. R. Ogden
CONTRACT: AF33(616)-7452, Battelle Memorial Inst.
ABSTRACT: Melting and the effects of other processing
variables on the quality and mechanical behavior of two
selected tantalum-base alloys have been studied. The two
alloy compositions selected, Ta-10Hf-5W and Ta-30Cb-7.5V,
were scaled up to 30-pound ingots and fabricated to sheet
using conventional techniques of extruding, forging, and
rolling. Mechanical-property data obtained from the sheet
in various conditions of heat treatment and after various
processing schedules show both alloys to possess good high-
temperature strength as well as low-temperature ductility.
Reproducibility of properties has also been demonstrated.

WADC TR 53-373 Sup 9

202

ASD TDR 62-11, OTS Release

March 1962

SUBJECT: IMPURITY ATOM-DISLOCATION INTERACTIONS
AND SUBSEQUENT EFFECTS ON MECHANICAL
PROPERTIES OF REFRACTORY METALS

ABSTRACT: Studies have been made of (a) the strain-aging behavior and (b) the effect of pre-straining and high temperature annealing on molybdenum crystals of two different purity levels prepared by electron beam melting. One batch of crystals contained an average of 20 ppm total interstitial impurities, the other an average of 250 ppm interstitials with the major constituent being carbon.

Yield point experiments revealed no strain-aging in either material in the "as-grown" condition, but after a high temperature anneal followed by rapid cooling the material containing carbon showed appreciable aging effects, and the purer material weak effects. The results are explained in terms of the low solid solubility of the interstitial elements in molybdenum under equilibrium conditions at moderate temperatures. The second series of experiments showed that pre-straining and annealing treatments which produce a sub-structure in molybdenum, also result in a strengthening of the material. The strengthening increases with increasing pre-strain and with increasing carbon content. The results indicate that sub-grain boundaries strengthen molybdenum in a qualitatively similar way to ordinary grain boundaries.

ASD TDR 62-56

January 1962

SUBJECT: THE DEVELOPMENT AND APPLICATION OF
POLYURETHANE COATINGS FOR HIGH REFLECTIVITY

INVESTIGATOR: A. S. Dalton

ABSTRACT: The objective of this paper is to present the importance of thermal radiation control in preserving the structural integrity of a weapon system and to outline the history of the investigation and definition of the problem, the research and development involved in solving it and the application engineering in adapting the solution to practical use. Polyurethane coatings have many desirable properties, show outstanding performance, and for this problem are better than any other coating investigated to date.

APPLICATION STUDIES

WADD TR 60-491

January 1961

SUBJECT: DEVELOPMENT OF REINFORCED CERAMIC MATERIAL SYSTEMS

INVESTIGATOR: L. M. Stejskal, et al

CONTRACT: AF33(616)-6511, Boeing Airplane Co.

ABSTRACT: The objective of the work covered by this report was to develop reinforced ceramic material systems capable of withstanding the effects of combustion products of various fuels at material temperatures to 4000°F, for use in advanced powerplant and vehicle applications. A number of ceramic compositions based on zirconium oxide and hafnium oxide were developed which satisfactorily withstood the 4000°F oxidizing and erosive environment. Techniques for fabricating these materials by trowelling and curing at low temperature, flame spraying, and hot pressing were developed. Stainless steel and molybdenum were used in various configurations to reinforce the ceramic materials. Small-scale components both reinforced and unreinforced, were fabricated and tested in the oxidizing and erosive environment at temperatures approaching 4000°F. These components were of configurations representative of nozzle ram-jet liners, and leading edges. This report describes the approach employed to screen, develop, evaluate, and refine the metal-ceramic composite material systems and components, and recommends the direction of future work in this area.

WADD TR 60-874

February 1961

SUBJECT: APPLICATION OF COMPOSITE CONSTRUCTIONS WITH HONEYCOMB AND FOAM CORES

INVESTIGATOR: W. T. Jackson

CONTRACT: AF33(616)-7322, Hexcel Products, Inc.

ABSTRACT: A method of foam-filling thin sections of aluminum honeycomb core was devised. Development of a process of bonding foam filled honeycomb cores in sandwich panels permitted testing of sandwich specimens in shear and compression. Synergistic strength was observed in foam stabilizing honeycomb core of nominal .0007" foil gauge but no synergism was evidenced in the nominal .002" and nominal .003" foil gauge foam-honeycomb composite cores. Increase in absolute strength of foam stabilized honeycomb varied directly with foam density and inversely with honeycomb cell size and foil gauge. Foam did not improve strength to weight ratio in the foam-honeycomb combinations tested. In view of this, the merits of these foam stabilized honeycomb cores are not in the areas of improved specific strength.

WADC TR 53-373 Sup 9

204

June 1961

SUBJECT: EVALUATION OF MATERIALS SYSTEMS FOR USE
IN EXTREME THERMAL ENVIRONMENTS UTILIZING
AN ARC-PLASMA-JET

INVESTIGATOR: J. W. Rosenbery, H. E. Smith, J. C.
Wurst

CONTRACT: AF33(616)-6198, University of Dayton

ABSTRACT: A small arc-plasma-jet facility was
modified for evaluating materials systems applicable to ex-
treme thermal environments. This facility was intended to
simulate end-use conditions as closely as possible without
requiring the use of large hyperthermal test chambers or
actual test firings. A "standard" test was established and
a group of materials representative of some typical thermal
protective systems was selected for evaluation. Suitable
fixtures and instrumentation for conducting these tests
were developed and utilized for a series of tests on various
materials systems at heat flux levels ranging from 100 to
540 Btu/ft²-sec.

Results, including weight loss, density
change, depth and volume of erosion, and front and back-face
temperatures, are presented for ablating, insulating, and
heat sink type materials systems.

ASD TN 61-102

SUBJECT: CURRENT VACUUM TECHNOLOGY AND PRACTICE

INVESTIGATOR: M. L. Minges

ABSTRACT: A review of vacuum technology literature
is presented. The review includes: (1) a general description
of vacuum system components, (2) an outline of the engineering
approach to vacuum system design, and (3) a discussion of
outgassing phenomena. This report is an introduction to the
vacuum technology field, however, a limited amount of quanti-
tative data is included in selected areas so that it may be
of value in preliminary vacuum system design.

ASD TN 61-117, Part I

August 1961

SUBJECT: FATIGUE OF METALS - ALUMINUM

INVESTIGATOR: Lt D. Ingels

CONTRACT: AF33(616)-7238, Belfour Engineering Co.

ABSTRACT: The graphs presented herein display
metals fatigue information from various sources of published
and unpublished test data which has been processed and re-

ASD TN 61-117, Part I (Continued)

generated through a semi-automatic data processing system. Each series or set of graphs contains descriptive information (legends) which identifies the material, test procedure, test conditions and the most significant test and/or material variables associated with the plotted data. The data displayed in each set of graphs is intended to answer very general "questions" and to serve as a guide to further investigation of specific areas within the subject presented.

ASD TR 61-181

September 1961

SUBJECT: A NEW CONCEPT FOR RE-ENTRY WINDOWS
INVESTIGATOR: S. Allinikov, F. W. Forbes
ABSTRACT: This window will allow good visibility including the re-entry period. The use of the insulating, slotted disc, disc rotation, and cooling the area between the disc and the window keeps the window at a moderate temperature. Conventional transparent material such as Pyrex may be used for the windows since the temperature maintained is moderate.

Visual photographic tests, feasibility tests, materials, and designs are discussed. The test environment was provided by a J-75 engine with afterburner, and the Aeronautical Systems Division's Hypersonic Temperature Gasdynamics Facility.

Optimum materials, designs, and possible applications are suggested.

ASD TR 61-207, OTS Release

May 1961

SUBJECT: CRACK STRENGTH AND CRACK PROPAGATION
CHARACTERISTICS OF HIGH STRENGTH METALS
INVESTIGATOR: R. Christensen, P. Denke
CONTRACT: AF33(616)-7444, Douglas Aircraft Co.
ABSTRACT: Results of a fracture testing program for high strength sheet metals are presented. More than 500 sheet stock panels ranging in width from one to 18" and in thickness from .020" to .100" were tested. These panels contained centrally located cracks which were generated principally by fatigue loading at various exposure times under a variety of environments. Fracture strength of the cracked panels were determined for rupturing temperatures

ASD TR 61-207 (Continued)

ranging from -340°F to 2500°F . The experimental results were studied analytically. A semi-empirical expression for residual strength is derived. This expression is based on a modification of the formula given by Crichlow for the effective width of the plastic zone. An expression for the rate of crack propagation also is presented. These formulas are shown to agree well with test results and are suitable for design applications. A digital analysis of the elastic and plastic stress and strain distribution in the cracked plate was performed. The analytical method is discussed and results are presented.

ASD TR 61-353

February 1962

SUBJECT: SURVEY AND FUTURE TRENDS OF GRAPHITE TECHNOLOGY
INVESTIGATOR: E. J. Dunn
CONTRACT: AF33(616)-6288, E. J. Dunn, Thomaston, Connecticut

ABSTRACT: A survey of the various graphites, in view of their advances during the past three years is presented. The various claims, properties, and processes of graphites are analyzed to assess their practicality, reliability, and reproducibility. The properties of graphite discussed and compared to aid the decision making designers in understanding the potential of graphites with regard to missile and space applications. Emphasis is placed on the need for more co-operation between the designer and materials engineer in view of the belief that graphites should not be treated as a "shelf" material, but as a family of unique materials. Likely trends in graphite development are pointed out and suggestions for advanced courses of development are mentioned.

ELECTRONICS

ASD TR 7-772(III)

December 1961

SUBJECT: DEVELOPMENT OF MANUFACTURING PROCESS
FOR HIGH PURITY ELECTRONIC CERAMICS
INVESTIGATOR: J. Battle, A. Marino, Jr.
CONTRACT: AF33(600)-42473, International Telephone
and Telegraph Corp.
ABSTRACT: The purpose of this project is to develop
new or improved manufacturing methods for large scale pro-
duction of ferroelectric and piezo-electric ceramic materials
such as titanates, zirconates, niobates and tantalates. A
major requirement is high purity (99.95%). The accomplish-
ments during this first period of Phase II have been the
selection of techniques for preparing barium metaniobate and
barium zirconate, and the construction of a chemical pilot
plant. (See ASD Interim Report 7-772 (II) - July 1961).
Synthesis of barium niobate by a direct metathetical reaction
and by the oxalate method has been accomplished. The re-
sulting products have been verified by X-ray diffraction
studies and chemically analyzed for purity. Barium zir-
conate has been successfully prepared by the oxalate method
while a suitable metathetical reaction is being sought.

ASD TR 7-772 (IV)

April 1962

SUBJECT: HIGH PURITY ELECTRONIC CERAMIC PROGRAM
INVESTIGATOR: A. J. Marino, Jr., J. Battle
CONTRACT: AF33(600)-42473, International Telephone
and Telegraph Corp.
ABSTRACT: The purpose of this project is to develop
new or improved manufacturing methods for large scale pro-
duction of ferroelectric and piezo-electric ceramic materials
such as the titanates, zirconates, niobates and tantalates.
A major requirement is high purity (99.95%). The
accomplishments during this second period of Phase II have
been the investigation of compatible barium zirconate tech-
niques, direct reaction of barium chloride and titanium
ester, chemical analysis techniques and the construction of
a chemical pilot plant. Synthesis of BaTiO_3 by the addition
of barium chloride directly to the organic ester in a
potassium hydroxide solution has been accomplished. Chelated
esters and other organic compounds are being investigated to
eliminate hydrolization during powder preparation.

ASD TR 7-838

June 1962

SUBJECT: REPRODUCIBLE THERMISTOR REFINEMENT
PROGRAM
INVESTIGATOR: M. C. Vanik, W. T. Barrett, J. E.
Herrera, et al
CONTRACT: AF33(657)-7104, W. R. Grace & Co.
ABSTRACT: Thermal instability of gold-doped mono-
crystalline silicon thermistors arose as a problem just
prior to the operation of the pilot line. Annealing and
improvements in the contact process should be the answer
to this problem.

CHEMICAL ENGINEERING

ASD TR 7-788(IV)

September 1961

SUBJECT: DEVELOPMENT OF MANUFACTURING METHODS FOR
GLASS FLAKE REINFORCED PLASTICS
INVESTIGATOR: K. R. Barr, L. J. Klahs, F. E. Manemeit,
A. D. Snyder
CONTRACT: AF33(600)-41885, Olin Mathieson Chemical
Corp.

ABSTRACT: A test manufacturing program has been initiated to determine feasibility of producing complex shaped laminates using glass flake reinforcement. A missile stabilizing fin and a missile nose cone insert were produced successfully in essentially a conventional commercial molding process. Flexural modulus of the flake reinforced fin was determined to be nearly three times that of a glass fiber reinforced structure now in production. Tensile and flexural strengths of the glass flake piece were lower than these characteristics in the fiber reinforced fin. The mechanisms of glass flake size reduction during mixing in a double-bladed mixer have been investigated, and high rates of degradation of the larger flakes were observed. Glass flake breakage during molding or dry blended laminates revealed a similar preferential degradation of large flakes during dry blend premixing. Ram extrusion of a polyester based glass flake premix (injection molding) was found to be unfeasible for thermosetting resin binders. Feed problems and uncontrollable curing of premixes in the sprue bushing of the unit resulted in poor laminations.

ASD TR 61-7-840

July 1961

SUBJECT: NUCLEAR HYDRAZINE PROGRAM
INVESTIGATOR: J. H. Cusack, R. I. Miller, H. P. Yockey
CONTRACT: AF33(600)-40878, Aerojet-General
Nucleonics

ABSTRACT: A new process is presented for accomplishing chemical reactions utilizing energy from a nuclear reactor in the form of fission fragment irradiation from suspended fissile materials. The inherent advantages, as well as the limitations of this new chemical process, are presented. The production of hydrazine from liquid ammonia was selected as a desirable demonstration of the fission fragment irradiation approach to this goal. Fully enriched uranium in the form of micron size uranium dioxide particles was suspended in liquid ammonia and exposed to a neutron flux

ASD TR 61-7-840 (Continued)

in the Livermore Pool Type Nuclear Reactor. Chemical and energy deposition analysis methods were developed and are described. Supporting effort in the fields of materials compatibility, fuel solubility and hazards analysis is discussed. Experimental equipment designs are presented together with detailed operating experience.

ASD TR 61-7-840A(III)

October 1961

SUBJECT: HYDRAZINE PROCESS DEVELOPMENT
INVESTIGATOR: J. Cusack, L. Carpenter, R. Miller, R. Pearson, F. Standerfer, H. Watanabe
CONTRACT: AF33(600)-42996, Aerojet-General Nucleonics
ABSTRACT: If fuel particle size degradation in an irradiation field becomes a serious processing problem, the fuel separation technique originally scheduled for use in the hydrazine program may be unsatisfactory. A boiling reactor concept, which would eliminate the fuel separation requirement, is being considered as an alternate approach. Until the extent of this problem is known, the experimental evaluation of hydroclone fuel separators is continuing. Gas chromatography analytical techniques have been developed to the point where detection limits for nitrogen, hydrogen, oxygen, and krypton are about 0.001 cc and hydrazine sensitivity is about 5 micrograms. Methods of lowering the hydrazine detection limit are being studied. A gamma ray sedimentometer has been designed for rapid particle size analysis of suspended fuels. Methods have been devised for the direct and indirect measurement of the average fission fragment energy escaping from particulate fuels. One method is based on the known yield of hydrogen from irradiated aqueous systems; the other depends on the direct measurement of fission fragment energy by means of a solid state detector.

ASD TR 7-840(V)

May 1962

SUBJECT: HYDRAZINE PROCESS DEVELOPMENT
INVESTIGATOR: R. I. Miller, R. L. Pearson, F. R. Standerfer
CONTRACT: AF33(600)-42996, Aerojet-General Nucleonics
ABSTRACT: The primary goal of this program is to develop, design, construct, and operate a continuous, in-reactor hydrazine production loop based on the fission-chemical process approach. This fourth quarterly progress

ASD TR 7-840A(V) (Continued)

report describes particulate fuel behavior in hydroclones, in filters, and in a flocculating environment. Alterations of the in-reactor loop, supporting components, and instrumentation, necessitated by a change in reactor facility are discussed. Equipment to be used for ammonia radiolysis, and for high energy compound and slurry radiation stability studies, are described.

ASD TR 7-880b(I)

December 1961

SUBJECT: SOLID PROPELLANT GRAIN CORE MANDRELS
INVESTIGATOR: D. F. Carey
CONTRACT: AF33(657)-7178, Thiokol Chemical Corp.
ABSTRACT: In commencing work on an Air Force contract for the development of a mandrel design technique for the forming of solid propellants, a literature survey of the properties of materials which might be used for this application was made. It is desired to develop mandrels which can be removed prior to firing. Recent advances in solid propellant rocket design has resulted in the need for mandrels capable of being removed through existing openings in the rocket motor case. The aluminum mandrels presently being used are unsatisfactory for this purpose. On the basis of the survey performed, segmented plastic foam, soluble salt bladders, and laminated plastic were determined to be the best design materials for fabrication of the mandrels.

FABRICATION

ASD TR 61-7-115

September 1961

SUBJECT: CERAMIC STACKED TUBE TYPE SN-2146C
INVESTIGATOR: S. L. Pawlikowski
CONTRACT: AF33(600)-37967, Sylvania Electric Products
ABSTRACT: Under the contract, a pilot line activity was continued on Type SN-2146C, a ceramic stacked tube, to improve the design, materials, manufacturing techniques and to evaluate the tube type under various environmental conditions. Tooling design changes necessary for attainment of maximum power output at minimum distortion were effected, the associated bell-jar exhaust equipment necessary for tube manufacture was installed, and life test ovens with temperature range capabilities of 300°C to 800°C were installed and evaluated. Parts processing and manufacturing techniques were evaluated and improved. Sealing techniques which insure a high degree of reliability were evolved. Methods of increasing production potential were satisfactorily developed and used. One hundred (100) good tubes of the final design were produced to meet the Air Force contractual requirements. An intensive tube evaluation program was carried out. This included tube testing under severe environmental conditions of shock, fatigue, vibration and evaluation of life test performance at elevated temperatures, as well as static and dynamic electrical tests. These data were analyzed and a final testing specification was evolved.

ASD TR 7-545 (I)

July 1961

SUBJECT: ELECTRO-DISCHARGE MACHINING PROGRAM
INVESTIGATOR: P. E. Berghausen
CONTRACT: AF33(600)-42282, Cincinnati Milling & Grinding Machines, Inc.
ABSTRACT: The first steps toward a comprehensive investigation of electro-discharge machining is presented with special emphasis upon optimization of process parameters. An industrial survey was conducted to isolate areas of greatest emphasis and to familiarize organizations with the program. Results of this survey are presented. Two electro-discharge machines have been designed and manufactured. Five power supplies have been analyzed and the data presented. Dielectric fluids of unconventional composition have been investigated and operating data tabulated. A fundamental investigation of the electro-discharge machining process has been undertaken and supporting experimental studies advanced. A concise theoretical treatment of the metal removal process is in progress.

May 1961

SUBJECT: TITANIUM DEVELOPMENT PROGRAM
INVESTIGATOR: C. W. Alesch, S. R. Carpenter
CONTRACT: AF33(600)-34876, General Dynamics
ABSTRACT: Ti-4Al-3Mo-1V alloy, because of its greater desirability for satisfying engineering properties requirements, greater freedom from incoming inspection difficulties, acceptability of mode of heat treatment and overall manufacturing capabilities is preferred over Ti-2.5Al-1.6V, Ti-6Al-4V, alloys for airframe construction employing solution heat treated and aged titanium alloys. In the supplemental limited evaluation of two additional titanium alloys, Ti-13V-11Cr-3Al was selected over Ti-5Al-2.75Cr-1.25 Fe due to its superior mechanical property values and spot weld strength characteristics. In the room temperature to 800°F range, the Ti-4Al-3Mo-IV alloy displayed the higher and more stable engineering strengths which combined with the superior creep resistance of the material contributed toward its selection for use in test part fabrication. At -100°F, all alloys display higher than room temperature strength but a tendency to borderline ductility is present. Metallographic surveys suggest that for other than martensitic structures, which tend to embrittle, the microstructures in mill product exert little influence on engineering properties. Strain effects tests, of laboratory specimens, show all alloys susceptible to Bauschinger effects and reflect the need for straining heat treatment for greater compression strength efficiency. These findings were confirmed by tests of material formed under shop production conditions at room temperature and elevated temperature conditions. Forming Ti-4Al-3Mo-1V and Ti-13V-11Cr-3Al at room temperature followed by aging gave best results. Forming Ti-5Al-2.75Cr-1.25 Fe at 600°F followed by aging showed best overall properties. Ti-4Al-3Mo-IV and Ti-13V-11Cr-3Al, spotwelded in the aged condition, satisfactorily met requirements of Specification MIL-W-6858A after exposure to elevated temperature for various time periods, whereas Ti-3Al-2.75Cr-1.25 Fe spotweld tests were marginal. The greater freedom of the Ti-4Al-3Mo-1V alloy from rejection cause, on one score or another upon "in-plant" receipt, asserted its production desirability. Heat treating characteristics of the Ti-4Al-3Mo-1V alloy indicated a good degree of fool-proofing in it to enhance its desirability over the other Ti alloys. Its mode of heat treatment was found acceptable to production although relatively long aging time presented schedule and equipment provisioning problems.

May 1961

SUBJECT: TITANIUM DEVELOPMENT PROGRAM
INVESTIGATOR: C. W. Alesch, S. R. Carpenter
CONTRACT: AF33(600)-34876, General Dynamics
ABSTRACT: Detailed incoming inspection data, inspection control chart data, creep test curves and curves comparing properties of various alloys are recorded herein. Interpretative discussions are found in Volume I.

May 1961

SUBJECT: TITANIUM DEVELOPMENT PROGRAM
INVESTIGATOR: A. P. Langlois, J. F. Murphy, E. D. Green
CONTRACT: AF33(600)-34876, General Dynamics
ABSTRACT: Ti-4Al-3Mo-1V alloy was found to be superior to Ti-2.5Al-16V and Ti-6Al-4V considering the overall forming and joining characteristics determined by the basic fabricability evaluation. In the supplemental evaluation of two additional alloys, the Ti-13V-11Cr-3Al was found to be superior to Ti-5Al-2.75Cr-1.25 Fe in the mechanical property values and spot weld strength characteristics. Ti-5Al-2.75Cr-1.25 Fe alloy was found to be more desirable from the forming standpoint. The use of Ti-13V-11Cr-3Al in the fully aged condition is considered undesirable from the fabrication standpoint. The material is brittle when heat treated to full strength values and demonstrates notch sensitivity to a greater extent than the other alloys evaluated. The most suitable sequence established to produce accurately formed detail parts of the selected solution treated Ti alloy was to form at room temperature in the solution heat-treated condition; hot size at the aging temperature for a controlled period; then furnace age. Joining the titanium alloys by resistance welding techniques should be accomplished in the fully heat treated condition to achieve the highest strength level. Fusion welding of these Ti alloys, on the other hand, should be performed in the solution heat treated condition to achieve highest mechanical property values. Cleaning of these Ti alloys prior to welding and elimination of rough sheared edges is very important in obtaining reproducible, high-quality joints. Machining tests establish that satisfactory drilling, countersinking and routing can be performed in either the solution treated or fully aged condition. In the latter case, tool life is reduced. Hand routing is, in general, impractical. Chemical milling proved feasible and necessary in the fully aged condition to avoid excessive hydrogen absorption. Vacuum degassing is practical, to remove hydrogen picked up during chem-milling

ASD TR 61-7-576, Vol III(Continued)

operations. The most suitable chemical cleaning method was a vapor-degrease, alkali clean and pickle sequence.

ASD TR 61-7-576, Vol IV

May 1961

SUBJECT: TITANIUM DEVELOPMENT PROGRAM
INVESTIGATOR: A. P. Langlois, J. F. Murphy, E. D. Green
CONTRACT: AF33(600)-34876, General Dynamics
ABSTRACT: The fabrication of complex parts for both present and future airframe units from Ti-4Al-3Mo-IV alloy can be achieved with proper design and consideration of alloy forming and joining limitations. The tail cone assembly was fabricated from Ti-4Al-3Mo-IV alloy on a gage for gage substitution basis to the available F-102A design drawings. This assembly was the largest of those selected for test in this program and represented all problems known to exist in airframe fuselage construction. Two wing leading edge configurations were fabricated from Ti-4Al-3Mo-IV utilizing a typical F-106 leading edge contour but with a constant section. Internal structure was so designed that fabrication could be achieved separately and assembly made by bolting, riveting and resistance spot welding all in the same test specimen. Two bulkhead assemblies, similar to F-102A tail cone attach bulkheads were fabricated from Ti-4Al-3Mo-IV alloy. The design was unique for the application of this Ti alloy. One assembly was riveted and the other was assembled by resistance spot welding. Three joining methods were evaluated to determine the most satisfactory production technique in the fabrication engine bleed air ducts similar to F-106 bleed air ducts. These were burndown flange, burndown and seam weld and burndown, rivet and braze. Compression and shear panels with and without core stiffeners were fabricated of Ti-4Al-3Mo-IV and Ti-13V-11Cr-3Al to ascertain the forming and joining problem areas. All panels when completed were in the fully aged condition.

ASD TR 61-7-576, Vol V

May 1961

SUBJECT: TITANIUM DEVELOPMENT PROGRAM
INVESTIGATOR: G. D. Lindeneau, D. H. Love, J. K. Neary, H. A. Buehler, G. F. Foelsch
CONTRACT: AF33(600)-34876, General Dynamics
ABSTRACT: Typical airframe structures, fuselage frames, wing leading edge, bleed air ducts, tail cone, shear

panels and compression panels of Ti-4Al-3Mo-1V and Ti-13V-11Cr-3Al were subjected to test loads in increasing increments of 100 degrees from room temperature to maximum temperatures of 800°F and 900°F, depending on the part. Riveted and resistance welded construction was evaluated in the fuselage frame and wing leading edge. Other components were either fusion welded, resistance welded, riveted or brazed. Components were subjected to static and repeated loadings with the exception of compression panels which had axial and side loads supplied. All components satisfactorily withstood static test loads. Under repeated load test, the resistance welded fuselage frame and wing leading edge, although adequate, did not perform as well as the riveted versions. Repeated load tests of resistance welded shear panels showed marginal results. Other components performed satisfactorily under repeated load conditions. Tests of spotwelded construction in the fuselage frame and wing leading edge demonstrated the need of large margins in spotwelded strengths at ends of members joined by spotwelding. Although Ti-4Al-3Mo-1V is not an optimum weldable alloy, the spotwelded assemblies of these components were considered adequate from repeated tests at elevated temperature, even though they did not perform as well as riveted construction. For example, the riveted fuselage frame withstood approximately 200% more repeated loads than the one of spotwelded construction. Air ducts in fusion welded, seam welded and riveted and brazed configurations satisfactorily withstood static and repeated test load requirements. The seam welded construction sustained the highest pressure in the burst tests. All resistance welded shear panels sustained design static test loads. The repeated load tests indicate that much more data is needed. The tests were not conclusive and fell short of expectations. Notch factors due to spotwelding need further investigation. Three types of compression panels in Ti-4Al-3Mo-1V and three types of compression panels in Ti-13V-11Cr-3Al withstood combined compression load and side load from pressure in excess of design loads. Panels in Ti-13V-11Cr-3Al exhibited a brittle type of failure-probably due to low elongation in the material.

ASD TR 61-7-632e

July 1961

SUBJECT: THERMAL SHOCK TESTING OF SCALED ALUMINA RADOMES
INVESTIGATOR: H. S. Pergament
CONTRACT: AF33(600)-37909, General Applied Science Laboratories, Inc.
ABSTRACT: Two scaled ceramic (98.5% alumina) radomes for future hypersonic interceptor missiles fabricated, using dry isostatic pressing techniques were thermal shock tested. Both radomes failed after following the scaled trajectory for about 20 seconds. Due to the complete destruction of the models, a determination of the cause of failure was not possible. A discussion of the tests of a stainless steel model, used to calibrate the methane heater and ceramic model, is included and the necessity for such tests explained. A significant conclusion reached as a result of these tests is that a high temperature methane heater has been developed which can be used to simulate the temperature distribution, heat flux and thermal stresses for a body in flight following an unsteady trajectory. An analytical investigation conducted to determine which of three possible trajectories was the most critical with regard to temperature and thermal stress is presented. Also included is a discussion of the design of the methane heater and a section on the mechanical design of the test facility.

ASD TR 61-7-773

May 1961

SUBJECT: NORTOBRAZE SYSTEM QUARTZ LAMP RADIANT HEAT BRAZING OF HIGH TEMPERATURE SANDWICH PANELS
INVESTIGATOR: H. Spector
CONTRACT: AF33(600)-40457, Northrop Corp.
ABSTRACT: The "Nortobraze" system has fabrication capability for brazing PH 15-7 Mo honeycomb structures. It is sufficiently versatile to handle edge members and densified core or other heat sinks while obtaining adequate strength properties. Curved and flat-to-conic sandwich panels can be made with a high degree of brazement reliability. Successful use of a moving radiant lamp heat source has been demonstrated for brazement and heat treatment of precipitation hardening steel honeycomb panels. The "Nortobraze" system can readily handle non-perforated core and high nickel content low thermal conductivity braze alloy. No evidence of nickel diffusion was found in any of the destructively tested panels. Modifying the sub-zero transformation cycle to -50°F for 30 minutes did not effect

the tensile strength properties of the faces or the honeycomb sandwich. The program had seven phases. The first four phases were designed to determine the limitations of the "Nortobraze" system in regard to simultaneous brazing and heat treating as a factor in design. These four phases investigated the brazing and heat treating of PH 15-7 Mo stainless steel panels of the following types:

- Flat with and without edge members, and with heat sinks
- Wedges up to 4 inches thick
- Single curvature with edge members
- Flat to conics with edge members

The brazed flat, single curvatures and flat to conic honeycomb panels can be made with a high degree of reliability with respect to brazements (filletting and node flow), within realistic tolerances and above minimum tensile physical properties. Tighter tolerances for the single curvature and flat to conic panels can be obtained by refinements in the cold cycle process. (Brazement of wedge honeycomb panels can be obtained with the "Nortobraze" system.) Approximately 55 honeycomb panels were fabricated for the first four phases; 21 of these were destructively tested. The last three phases of this program were designed to determine the feasibility of continuous zone brazing and heat treating of PH 15-7 Mo stainless steel honeycomb. Flat, wedge and single curvature panels were investigated. Careful temperature programming of the moving quartz lamp source is required to minimize initial panel buckling caused by thermal shock. Approximately 17 panels were fabricated for these three phases. Seven panels were used for equipment checkout and feasibility tests and four panels were destructively tested. The results of the destructive tests compared very well with those obtained in the first four phases.

ASD TR 7-778(v)

December 1961

SUBJECT: DEVELOPMENT OF MANUFACTURING METHODS FOR
GLASS FLAKE REINFORCED PLASTICS
INVESTIGATOR: K. R. Barr, L. J. Klahs, F. E. Manemeit,
A. D. Snyder
CONTRACT: AF33(600)-41885, Olin Mathieson Chemical
Corp.

ASD TR 7-778(V) (Continued)

ABSTRACT: Investigation of the suitability of glass flake reinforced resin systems in commercial molding operations has been continued with the fabrication of a third shape by compression molding, and two parts by transfer molding processes. These developments demonstrate two major problems yet to be overcome to provide high quality, high strength laminates. Glass flake degradation and poor control of resin flow characteristics have prevented the fabrication of parts having superior qualities than presently available in conventional molding compounds. Thickness studies have yielded molding data and physical characteristics on slabs ranging from 1/8 inch to 2 inches in cross section. Casting of glass flake premixes in high speed horizontal centrifugal casting equipment has begun. Development of a solvent coating technique is being attempted to make use of solid resins having higher inherent strengths than those systems used to date.

ASD TR 62-7-799

March 1962

SUBJECT: MANUFACTURING METHODS FOR INSULATED AND COOLED DOUBLE-WALL STRUCTURES
INVESTIGATOR: E. C. Haight
CONTRACT: AF33(600)-40100, Bell Aerosystems Co.
ABSTRACT: This Section II of the final technical engineering report, describes the program, and presents the results of the static load and thermal tests conducted on the Double-Wall end-item at Wright-Patterson Air Force Base, Ohio. In addition, it describes the thermal cycling tests conducted on a molybdenum outer wall panel to a maximum temperature of 2600°F.

ASD TR7-827 (II)

October 1961

SUBJECT: TUNGSTEN SHEET ROLLING PROGRAM PHASE II
INVESTIGATOR: W. J. Schoenfeld
CONTRACT: AF33(600)-41917, Universal-Cyclops Steel Corp.
ABSTRACT: Phase II of this program - Ingot Process Development, has been completed. Parameters of investigation included powder chemistry, electrode manufacture, electrode assembly, vacuum arc melting, and initial breakdown techniques. For the initial work in this phase, standardized electrode specifications were developed. Preliminary melting investigations, studying the effects of electrode to mold

ASD TR 7-827 (II) (Continued)

ratio, provided an increase in yield of approximately 12% over previously established melting conditions. Continued melting provided ingots for destructive ingot evaluation and initial breakdown investigations. Direct forging and extrusion techniques were utilized in initial breakdown. Direct forging was essentially unsuccessful but extrusion to rounds with subsequent press forging to sheet bar and direct extrusion to sheet bar were very successful.

ASD TR 62-7-854

February 1962

SUBJECT: HIGH TEMPERATURE INFRARED WINDOW MANUFACTURE BY HIGH ENERGY FORMING
INVESTIGATOR: D. E. Sheffer, E. A. Bush
CONTRACT: AF33(600)-41904, General Dynamics/Pomona
ABSTRACT: The technical feasibility of using the Dynapak machine method of producing high-energy-rate forces to form ceramic powders into virtually nonporous bodies for application as high temperature windows is demonstrated. Transparent compacts 2 inches in diameter and one-fourth inch thick of potassium bromide, potassium chloride, sodium chloride and lithium fluoride were formed. Barium fluoride, magnesium fluoride, and zinc sulfide were produced as transparent pieces with fair transmission in the infrared. Tooling for compaction operations at temperatures up to 1100°F is described. An analysis of the dynamics of the Dynapak machine as applied to the compaction process is presented.

ASD TR 7-858(I)

July 1961

SUBJECT: LARGE PLASTIC ROCKET MOTOR CASES
INVESTIGATOR: J. R. Turner, F. W. Dallon, W. G. Morse
CONTRACT: AF33(600)-42511, Thiokol Chemical Corp.
ABSTRACT: The design, study and vendor evaluation phase of a program to develop manufacturing methods, control, equipment and processes expressly directed toward the fabrication of large size, fiberglass reinforced, solid propellant rocket motor cases has been completed. Design modifications resulting from Phase I activities include: (a) an all fiberglass skirt with metal inserts; (b) use of elastomeric materials for internal insulation and of graphite-phenolic insulator rings at each port as a thermal insulative barrier; (c) a new blast tube design; and (d) use of a collapsible or destructible mandrel for propellant

ASD TR 7-858 (I) (Continued)

loading, which makes possible a more efficient unit. The application of improved material formulations and advances in component fabrication techniques is contributing measurably to design finalization. Computer programmed investigations of design parameters contributed to the decisions affecting the acceptance of some, and the rejection of others, of the concepts originally proposed. Above are the major accomplishments of Phase I of AF Contract 33(600)-42511, Large Plastic Rocket Motor Cases. Below is a detailed discussion of the items mentioned. An all-fiberglas skirt with metal inserts at the attachment points will be used in lieu of the all-steel skirt originally proposed. Elastomeric materials will be utilized for internal insulation and graphite-phenolic insulator rings at each nozzle port will serve as a thermal insulative barrier between the graphite throat and the ablative elastomeric insulation.

ASD TR 61-7-876

October 1961

SUBJECT: A STUDY OF FORGING VARIABLES
INVESTIGATOR: H. J. Henning, R. J. Carlson, A. M. Sabroff, F. W. Boulger
CONTRACT: AF33(600)-42963, Battelle Memorial Inst.
ABSTRACT: A survey of the present state of the art of forging was made to evaluate the influence of metal properties and processing variables on forgeability and to determine the applicability of present theories of metal deformation to commercial forging operations. The physical and mechanical properties, such as melting point, crystal structure, flow stress, ductility, etc., that determine the range of conditions necessary for successful forging are discussed. Limitations on the range of forging conditions imposed by final mechanical-property requirements, such as tensile strength, fatigue strength, impact strength, etc., also are reviewed. Experimental forging studies are planned to provide additional data on the effects of such variables as forging temperature, deformation rate, amount of deformation, size, and configuration. Both open- and closed-die forging trials will be made on three types of alloys.

ASD TR 61-7-868

October 1961

SUBJECT: FABRICATION OF CUTTING TOOLS BY ELECTROPHORETIC DEPOSITION
INVESTIGATOR: M. H. Ortner, K. A. Gebler

ASD TR 61-7-868 (Continued)

CONTRACT: AF33(600)-41436, Vitro Corp.
ABSTRACT: The technical feasibility of fabricating carbide coated cutting tools by the electrophoretic deposition process has been demonstrated. Carbide coated lathe tool inserts were prepared which were equal or superior in tool life to commercial, solid, Type-C-6 carbide lathe tools in turning AISI 4340 steel. The addition of small amounts of boron to the cementing metal proved beneficial in lowering the sintering temperature of Grades C-2, C-6, and C-5 carbide coatings on a variety of substrates. A significant feature of these coated tools is the development of a well-defined, hard diffusion zone between the substrate and the carbide coating.

ASD TR 7-888(I)

September 1961

SUBJECT: DEVELOPMENT OF ULTRASONIC WELDING EQUIPMENT FOR REFRACTORY METALS
INVESTIGATOR: J. B. Jones, N. Maropis, C. F. DePrisco, J. G. Thomas, J. Devine
CONTRACT: AF33(600)-43026, Aeroprojects, Inc.
ABSTRACT: Joining refractory and superalloy metals in thicknesses up to 0.10 inch by ultrasonic welding is feasible. A first approximation of the acoustical energy required indicates that the requisite welding equipment is also feasible. This report is a compilation and discussion of information pertinent to the development of ultrasonic welding equipment for joining AM-355 steel, Inconel X, Rene' 41, tungsten, molybdenum-0.5% titanium, and columbium alloy (DuPont D-31). The feasibility of welding the materials and gages of interest is supported by data, appropriately referenced, from previous work with thinner material. Information on transducer, coupler, and tip materials is presented with information on evaluating efficiency and practicability. Design information on spot-type and roller-seam welding machine tips is presented. Data on various properties of the weldment materials are tabulated.

ASD TR 7-884(I)

July 1961

SUBJECT: SUBMICRON SIZED PARTICLES
INVESTIGATOR: J. D. Holmgren, J. O. Gibson, R. Weidman
CONTRACT: AF33(600)-42916, Vitro Corp.
ABSTRACT: Work was initiated on the preparation of high purity submicron size powders of refractory and non-

ASD TR 7-884(I) (Continued)

refractory materials in the 1000 to 100 Angstrom size range. Sample quantities of various submicron refractory oxides were prepared using the arc vaporization technique. Preliminary testing was started for application of this technique for preparation of submicron size refractory metal and carbide powders. Surveys were carried out to determine the state-of-the-art for the production of submicron size powders. When the survey results are complete, the data will be analyzed to establish the relative advantage of the various processes for economical production of high purity submicron materials.

ASD TR 7-884(II)

October 1961

SUBJECT: SUBMICRON SIZED PARTICLES
INVESTIGATOR: J. D. Holmgren, J. O. Gibson, R. Weidman
CONTRACT: AF33(600)-42916, Vitro Corporation
ABSTRACT: Equipment modification and a state-of-the-art survey have been completed. Significant progress has been made toward the development of arc vaporization techniques for a number of refractory materials and, in several cases, extensive characterization and analysis of representative batches are underway. Process development studies have been started on various refractory oxides, metals, and carbides including silica, alumina, thoria, tungsten oxide, iron oxide, titanium oxide, magnesium oxide, zirconium oxide, tungsten metal, aluminum metal, tantalum carbide and tungsten carbide. Tentative processes have been established for a number of the oxide systems and oxide products in the 130-160 Å particle size range have been prepared. The technique has been extended to the metal and carbide systems to prepare samples of highly pyrophoric products ranging down to 42 Å in particle size.

ASD TR 7-910 (II)

November 1961

SUBJECT: EFFECTS OF ANDROFORMING ON MATERIAL PROPERTIES
INVESTIGATOR: C. D. Lantz, C. B. Crandall, R. E. Johnson, C. F. Morris
CONTRACT: AF33(600)-42847, General Dynamics
ABSTRACT: The sheet metal forming parameters of the Model J Androform machine were established for various heat treated conditions of aluminum, stainless steel, and titanium. Nomographs were derived for determining the

longitudinal and transverse contour combinations which are within the Androform machine forming limits. Mechanical property tests were conducted. The machine forming limits were derived for each material and material gage by progressively "tightening" the machine adjustments until a maximum useable contour was obtained. Statistical experiments were designed to derive the full forming range of the machine with a limited number of parts formed on selected machine set ups. The longitudinal and transverse contours of parts formed from various alloys and gages were measured. Equations were statistically derived to predict the contours that would result from other machine settings. Two types of nomographs were developed from the equations. The Androform process was theoretically analyzed utilizing fundamentals of the theory of plasticity. The stresses and strains obtained from this analysis were then correlated to contour measurements obtained from the actual forming tests.

December 1961

SUBJECT: STUDY OF MANUFACTURING PROCESSES AND DEVELOPMENT OF MANUFACTURING METHODS TO PROVIDE HIGH TEMPERATURE TIRES

INVESTIGATOR: S. van der Burg, S. J. Nelson, J. G. Manchetti

CONTRACT: AF33(600)-42970, United States Rubber Company

ABSTRACT: Butyl, chlorobutyl, ethylene propylene rubber and a polyester type polyurethane have been selected for detailed compounding studies for use as high temperature rubber compounds. Resin cured butyl, ethylene propylene rubber cured with Dicap and low sulfur, and polyurethane cured with Dicap, and chlorinated butyl rubber have the best potential for high temperature aircraft tires. In resin cured butyl, SAF black, compared to ISAF, HAF, and MPC blacks had the best room temperature and high temperature strength. Both the conventional nylon-natural rubber and belted radial steel-ply natural rubber belted radial steel ply-natural rubber designs are adequate, in the testing thus far, with respect to the room temperature test requirements. Dynamic testing of tires was initiated to determine the adequacy of current designs to the room temperature contract requirement requirements.

ASD TR 7-924 (II)

May 1962

SUBJECT: FIBER REINFORCEMENT OF METALLIC AND
NONMETALLIC COMPOSITES
INVESTIGATOR: R. H. Baskey
CONTRACT: AF33(657)-7139, Clevite Corp.
ABSTRACT: Parameter studies are in progress to de-
termine the effect of fiber diameter, fiber length, fiber
spacing, fiber direction and fiber-to-matrix bond strength.

The fibers and powdered metal have been consolidated by hot pressing, hydrostatic pressing or cold pressing followed by sintering.

ASD TR 7-937

June 1962

SUBJECT: MANUFACTURING METHODS AND DESIGN PRO-
CEDURES FOR BRAZED REFRACTORY METAL
HONEYCOMB PANELS
INVESTIGATOR: J. W. McCown, C. R. Wilks, L. J. Gagola,
H. H. Hotchkiss, M. M. Schwartz, J. E.
Miller
CONTRACT: AF33(657)-7276, Martin Marietta Corp.
ABSTRACT: Brazed honeycomb sandwich panels using
molybdenum and columbium core and facings provide lightweight
structural coverings for high temperature applications on
aerospace vehicles. Panel configurations selected for
fabrication simulate a hot structural and a radiant heat
shield application. Manufacturing processes and procedures
developed in the program will be used to fabricate test
panels. Elevated temperature tests will determine thermal
and structural capabilities of the test panels.

BASIC INDUSTRY

ASD TR 61-7-661

May 1961

SUBJECT: DEVELOPMENT OF HIGH TEMPERATURE RADIANT
GAS BRAZING METHOD FOR HONEYCOMB PANELS
INVESTIGATOR: W. C. Troy
CONTRACT: AF33(600)-38317, Solar Aircraft Co.
ABSTRACT: An effective low cost radiant gas method
for brazing honeycomb sandwich has been developed using
small radiant burners. Thermal control and configurational
flexibility inherent in the new brazing method permits great
simplification in high temperature tooling, leading to lower
product costs. In addition, the development of simplified
panel assembly procedures provides a process which consistently
furnishes metallurgically sound components from PH 15-7Mo
and A-286 which normally braze with difficulty. An extensive
program of mechanical tests evaluated the quality of the
honeycomb sandwich panels.

The radiant gas heating device used, employed 224 small burners manifolded into 14 groups for zone temperature control. A total of 77 full-size panels (20 by 54 inches) brazed in the equipment verified the effectiveness of the heating method on a production scale. These panels in turn furnished specimen material for over 400 static and dynamic tests of honeycomb sandwich, in addition to several hundred quality control tests. A new brazing procedure was developed for brazing A-286 to overcome difficulties inherent because of the Ti and Al content of the alloy, and development of a special process was necessary to control gravity flow of the silver-base alloy used for brazing PH 15-7Mo.

ASD TR 61-7-700

March 1961

SUBJECT: DEVELOPMENT OF 10⁷ GYRO SPIN AXIS
BEARINGS
INVESTIGATOR: J. T. Luxon, H. S. Kontrovitz, R. P.
Wolfe, P. A. Simmons
CONTRACT: AF33(600)-38120, General Motors Corp.
ABSTRACT: This project was initiated to assure the
availability of 10⁷ gyro spin axis bearings to meet the
scheduled requirements of this inertial guidance system.
This was to be accomplished by improving the bearing life and
capability to reproduce. The program goals of 80 percent
acceptability through a life (1) of 500 hours were achieved

ASD TR 61-7-700 (Continued)

by an improvement in design details. Functional testing indicates that these bearings may operate successfully in the gyro but final verification was not included in this contract. The modifications in bearing design were restricted to those which retained dimensional compatibility with the present gyro wheel. The basic 107 design was altered to incorporate improved separator material, shields, tapered lands and grooved O.D. separators. The second major goal of this contract was the development of new manufacturing and gaging techniques and equipment to produce bearings with all critical tolerances held to 20 to 40 micro-inches. The goal was reached. A specification has been prepared covering the design modifications and released as a separate document prior to publication of this report.

ASD TR 61-7-787a

October 1961

SUBJECT: WIDE, CLOSE-TOLERANCE STEEL SHEETS
INVESTIGATOR: B. B. Moss
CONTRACT: AF33(600)-42793, Douglas Aircraft Co., Inc.
ABSTRACT: Six major aerospace industry and aircraft companies entered into contracts with the U. S. Air Force to evaluate wide, close-tolerance steel sheets produced by the U. S. Steel Corp. by the Sandwich Rolling Process. The steels rolled included AISI 4340, H-11, AM 350, PH 15-7Mo, and A-286. The evaluation covered wide sheet handling, inspection, fabrication, and testing. Each participant presented the results of his evaluation at a symposium held at Pacific Palisades, 27 and 28 June 1961. The preparation and discussion which followed form the body of this report. The data presented indicate that side steel sheets were produced with satisfactory metallurgical properties and to tolerances approximately half of the present standard width tolerances. Large diameter missile forward closures and aircraft bulkheads were formed from wide single sheets by explosive forming and shear forming. Missile motor cases which tested satisfactorily were produced by the roll-weld technique, from wide steel sheets using a single sheet in a 360° wrap.

ASD TR 7-816

September 1961

SUBJECT: DEVELOPMENT OF NON-DESTRUCTIVE INSPECTION
TECHNIQUES FOR ANTI-FRICTION BEARINGS
INVESTIGATOR: R. A. Baughman

ASD TR 7-816 (Continued)

CONTRACT: AF33(600)-41676, General Electric Co.
ABSTRACT: The statistical nature of bearing life caused by metallurgical defects, such as inclusions, have a major influence on bearing design. As a result of development work completed, a new method of ultrasonic inspection is now available so that non-destructive inspection for these defects in bearing races can be improved. Using this technique it was possible to screen out simulated bearing specimens having shorter lives, leaving a balance of specimens, none of which failed prematurely.

ASD TR 7-904 (I)

October 1961

SUBJECT: STRUCTURAL FABRIC PROGRAM
INVESTIGATOR: J. T. Harris, J. O. Miller
CONTRACT: AF33(600)-43036, Goodyear Aircraft Corp.
ABSTRACT: The purpose of this program, as related to aerospace applications, is to provide a means of manufacturing large low-density AIRMAT structures made of metallic cloth and yarns capable of small volume packaging. The work reported on herein was accomplished during the first quarterly period under Contract AF33(600)-43036. This effort encompasses an industrial survey of loom manufacturers, a study of process variables and the selection of a textile consultant. The aforementioned efforts are directed toward the development of a loom procurement specification and the actual procurement of a loom capable of producing a low density AIRMAT in the order of 10 to 20 feet wide with a maximum depth of eight (8) feet.

ASD TR 62-7-546

April 1962

SUBJECT: CLOSE TOLERANCE STEEL FORGING DEVELOPMENT
INVESTIGATOR: C. E. Proffitt, H. C. Thomas
CONTRACT: AF33(600)-36659, Boeing Airplane Co.
ABSTRACT: A manufacturing process was developed to produce improved high strength thin walled steel parts for mission oriented systems forged to their final configurations. Three steel alloys, AISI 4340, 300M, and AISI-SAE H-11, were successfully forged to an intricate interrupted H-beam shape. Comparative testing of machined and close tolerance forged parts showed approximately 10 percent greater ultimate strength and 40 percent greater fatigue life for the close tolerance forgings.

January 1962

SUBJECT: TITANIUM DIRECTIONALITY PROGRAM
INVESTIGATOR: A. E. Leach
CONTRACT: AF33(600)-37938, Crucible Steel Co.
of America

ABSTRACT: This manufacturing process development determined techniques for strip processing to minimize high directional mechanical properties in three DOD titanium alloys. Full-scale strip processing production operations starting with 4000 pound Ti-6Al-4V, Ti-4Al-3Mo-1V, and Ti-2 1/2Al-16V ingots have shown that the Ti-2 1/2Al-16V alloy is almost ideally suited to strip processing, developing negligible directionality and having excellent rolling and processing characteristics. The production of Ti-2 1/2Al-16V sheet by strip rolling instead of hand sheet processing will result in greater economies in production of better gage, flatness, and surface finish control.

II. APPENDIX, INVESTIGATOR INDEX

	Page
Aarnes, M. M. -----	199
Abbott, G. L. -----	179
Abbott, N. J. -----	121
Abramson, H. N. -----	160
Adams, A. A. -----	45
Adams, M. R. -----	155
Aftergut, S. -----	172
Aichroth, J. -----	167
Albrecht, B. -----	48
Alesch, C. W. ----- 214,	215
Allen, E. R. -----	64
Allen, V. R. -----	81
Alley, C. W. -----	68
Allinikov, S. -----	206
Alvares, N. J. -----	179
Anderson, G. -----	37
Anderson, L. G. -----	58
Andrew, K. F. -----	14
Anthony, F. M. ----- 192,	193
Apodaca, D. R. -----	44
Atlas, L. M. -----	59
Atwell, W. H. -----	70
Backer, S. ----- 119,	120
Bacon, R. -----	7
Bailar, J. C., Jr. ----- 80,	90
Baker, G. S. -----	4
Bambenek, R. A. -----	122
Baranauckas, C. F. -----	79
Barr, K. R. ----- 210,	219
Barrett, W. T. -----	209
Barsness, D. A. -----	138
Bartkowiak, A. -----	133
Baskey, R. H. -----	226
Batchelor, J. -----	109
Battle, J. -----	208
Baughman, R. A. -----	228
Baum, G. -----	77
Baun, W. ----- 187,	188
Beatty, G. H. -----	202
Beauchamp, E. K. -----	4
Beaver, W. W. -----	54
Beck, F. H. -----	36
Behnke, F. W. -----	189
Behun, J. D. -----	136

	Page
Bell, N. W. -----	183
Belser, R. B. -----	61
Bennett, D. G. -----	101
Benson, D. -----	20
Benton, E. R. -----	128
Berg, D. -----	157
Berghausen, P. E. -----	219
Berkowitz, A. E. -----	160
Berkowitz, J. -----	10
Berry, G. C. -----	81
Bicelli, L. P. ----- 21, 23,	25
Blackburn, P. E. -----	14
Blatherwick, A. -----	50
Blessing, A. H. -----	193
Blesto, N. -----	96
Bloomfield, J. J. -----	89
Bohner, G. E. -----	74
Boller, K. H. ----- 113,	117
Bond, W. C., Jr. -----	85
Booker, J. -----	3
Born, J. W. -----	70
Borsanyi, A. S. ----- 132,	133
Bosmajian, G. -----	123
Boulger, F. W. -----	222
Bowie, G. E. -----	50
Boyd, K. E. -----	150
Boyd, W. K. -----	51
Bradbury, E. J. -----	89
Bradley, D. C. ----- 73,	82
Brady, A. P. -----	115
Braid, S. -----	140
Brand, J. A. -----	17
Brande, H. ----- 131,	132
Brassart, F. A. -----	14
Bratt, L. -----	72
Braude, H. -----	134
Bredzs, N. -----	54
Breiter, M. -----	26
Brisbane, A. W. -----	38
Brotherton, R. J. -----	77
Brown, C. B. -----	30
Brown, G. P. ----- 72,	86
Brown, H. C. -----	71
Brown, R. D. -----	150
Brownfield, C. D. -----	44
Brumfield, P. E. ----- 82,	90
Bryant, P. J. ----- 141,	156
Buckley, W. H. -----	193
Bueche, F. -----	80

	Page
Buehler, H. A. -----	216
Buhler, R. D. -----	116
Burton, R. A. ----- 148, 149,	150
Bush, E. A. -----	221
Butler, G. B. -----	85
Butner, M. F. -----	148
Callihan, F. R. -----	143
Caminitz, H. C. ----- 101,	111
Cape, J. -----	175
Caramante, D. E. -----	109
Carey, D. F. -----	212
Carithers, M. D. -----	61
Carlson, R. D. -----	79
Carnahan, D. R. -----	48
Carlson, R. J. -----	222
Carpenter, L. -----	211
Carpenter, S. R. ----- 214,	215
Carrigan, R. -----	172
Cart, E. N. -----	148
Cartledge, F. K. -----	70
Casassa, E. F. -----	81
Cavell, E. A. S. -----	87
Cercao, J. M. -----	170
Chamberlain, D. ----- 72,	115
Chandhok, V. K. -----	11
Chapin, E. C. -----	83
Charles, M. -----	171
Cheathan, R. G. -----	120
Cherry, W. H. -----	159
Chessick, J. J. -----	147
Chick, B. -----	37
Childers, H. M. -----	170
Chlystek, S. J. -----	187
Christian, J. B. -----	152
Christensen, D. -----	116
Christensen, R. -----	206
Chu, C. C. ----- 120,	127
Chu, W. -----	160
Churchill, A. V. -----	134
Clark, B. F. -----	75
Clark, J. W. -----	56
Cody, G. D. -----	159
Coldren, A. P. -----	48
Coleman, D. G. -----	108
Conners, C. R. ----- 10,	65
Connolly, J. W. ----- 180,	181
Conroy, J. R. -----	92
Cook, J. C. -----	160

	Page
Coplan, M. J. -----	127
Coskren, R. J. -----	120
Costello, G. -----	33
Coth, R. -----	18
Crandall, C. B. -----	224
Cresci, R. J. ----- 175,	176
Cuellar, J. P. -----	68
Cuff, F. B. -----	16
Cusack, J. H. ----- 210,	211
Dakin, T. W. -----	157
Dale, J. W. -----	83
Dallon, F. W. -----	221
Dalton, A. S. -----	203
Daniel, B. ----- 153,	154
Daniel, I. M. -----	108
Das, Y. C. -----	31
Delphenich, J. S. -----	94
Denke, P. -----	206
DePierre, V. -----	15
DePrisco, C. F. -----	223
Devine, J. -----	223
Devins, J. C. -----	165
Dewhirst, D. L. -----	35
Dharmarajan, S. -----	32
Dick, B. G., Jr. -----	4
Dioguardo, P. R. -----	201
Divens, W. C. -----	157
Dolloff, R. T. -----	9
Dorn, J. E. -----	44
Doughty, L. E. -----	100
Douglass, C. A. -----	6
Downer, T. M. -----	190
Doyle, C. D. -----	86
Doyle, W. D. -----	160
Draper, R. N. -----	8
Driscoll, J. L. -----	132
Drowart, J. -----	2
Duckworth, W. -----	5
Dukes, W. H. ----- 192,	193
Duletsky, P. S. -----	15
Dulis, E. J. -----	11
Dunn, E. J. -----	207
DuPont, F. Y. -----	165
Durelli, A. J. -----	165
Dzuna, E. R. -----	141

	Page
Eaton, D. C. G. -----	51
Ebcioğlu, I. K. -----	41, 104
Eckert, A. C. -----	46
Economos, C. -----	171
Edwards, H. -----	131, 132
Ehlers, G. F. L. -----	77, 88
Elbaum, C. -----	37
Elliot, R. G. -----	92
Ellis, R. B. -----	124
Elsner, N. B. -----	55
Emrick, J. E. -----	22
Engel, O. G. -----	19
Engholm, G. -----	122
Epstein, G. -----	107
Euston, C. -----	186
Fabuss, B. M. -----	132, 133
Falk, R. A. -----	143
Farmer, R. W. -----	110
Favor, R. J. -----	41, 202
Feigl, H. -----	26
Fenske, M. R. -----	130, 144
Fessett, D. J. -----	146
Fiel, F. -----	103
Fieldhouse, I. B. -----	177, 193
Findl, E. -----	131, 132
Findley, W. -----	37
Fink, F. W. -----	51
Fischer, D. -----	187
Foelsch, G. F. -----	216
Fopiano, P. J. -----	24
Forbes, F. W. -----	206
Ford, F. -----	74
Ford, R. A. -----	43
Fox, R. B. -----	76
Fox, T. G. -----	81
Frank, A. J. -----	183
Freeman, J. W. -----	48, 196, 200
Freestone, W. D. -----	127
Freudenthal, A. M. -----	39, 43, 48
Frey, D. A. -----	89
Freyer, W. -----	78
Fruendlich, M. M. -----	142
Fu, C. C. -----	34, 52
Gadd, J. D. -----	63, 64
Gadkary, S. D. -----	80
Gagola, L. J. -----	226
Gardiner, K. W. -----	190

	Page
Gebler, K. A. -----	222
Gehrke, J. H. -----	14
Geiselman, D. -----	20
Gerard, G. -----	29, 30
Gerry, H. T. -----	68
Gettings, J. F. -----	189
Gibbs, P. -----	4
Gibson, J. O. -----	223, 224
Gideon, D. N. -----	41
Gilman, H. -----	70
Gittleman, J. I. -----	159
Glaser, P. E. -----	10, 129
Gluck, J. V. -----	196, 200
Goforth, R. E. -----	43
Goldman, A. -----	86
Goldschmidt, H. J. -----	17
Goodman, L. E. -----	30, 31
Gourdine, M. C. -----	137
Gravina, A. -----	170
Gray, T. J. -----	20
Graziano, M. -----	21, 23
Green, E. D. -----	215, 216
Green, J. -----	95
Green, R. O. -----	14
Greenhaus, H. L. -----	165
Grigger, J. C. -----	67
Grindle, S. -----	116
Groll, D. S. -----	40
Grover, H. J. -----	41
Gudzinowicz, B. J. -----	132
Gulbransen, E. A. -----	14
Gutfreund, K. -----	108
Haber, H. S. -----	190
Haight, E. C. -----	220
Halle, H. -----	173, 174
Handlovits, C. E. -----	91
Harmsworth, C. L. -----	67
Harrah, L. A. -----	180, 184
Harris, E. E. -----	79
Harris, J. T. -----	229
Harrison, W. -----	169
Hauser, C. F. -----	85
Hauser, R. L. -----	97
Hayes, J. E. -----	41
Hayford, A. W. -----	68
Headrick, R. E. -----	99, 105
Hedenburg, J. F. -----	141

	Page
Heise, R. E. -----	12
Heller, R. -----	34
Helminiak, T. E. -----	80
Henning, H. J. -----	222
Henning, R. G. ----- 195,	200
Herrera, J. E. -----	209
Herron, A. M. -----	126
Hickman, W. R. -----	92
Hill, W. H. -----	31
Hirt, H. C. -----	62
Hoeve, C. A. J. -----	81
Hogle, D. H. -----	157
Hokanson, H. A. -----	26
Holmgren, J. D. ----- 223,	224
Hopkins, V. -----	144
Horn, F. H. -----	159
Hotchkiss, H. H. -----	226
House, P. A. ----- 93,	98
Howse, P. T. -----	176
Huggins, R. A. -----	47
Hulbert, S. F. -----	20
Hyde, C. -----	5
Iannucci, A. -----	45
Imai, M. -----	141
Imgram, A. G. -----	192
Ingles, D. ----- 197,	205
Isham, A. B. -----	109
Islinger, J. S. -----	108
Issacs, L. -----	76
Jackson, J. D. -----	51
Jackson, W. T. -----	204
Jacobs, I. S. -----	164
Jaffe, H. H. -----	84
Jaffe, N. -----	44
Jaffee, R. ----- 15,	192
Jagodowski, S. S. -----	142
Jakobsen, R. J. -----	188
Janssen, J. -----	25
Jefferys, R. A. ----- 63,	64
Jenkins, R. J. -----	180
Joerg, P. -----	116
Johns, I. B. -----	83
Johnson, D. E. -----	128
Johnson, D. R. ----- 165,	178
Johnson, J. -----	160
Johnson, R. E. -----	224
Johnson, V. J. -----	158

	Page
Johnston, R. K. -----	68
Jones, D. E. G. -----	40
Jones, J. B. -----	223
Kan, P. T. -----	136
Kandler, L. -----	26
Kasak, A. -----	11
Kaswell, E. R. -----	121, 127
Katz, M. -----	170
Kaufman, L. -----	9, 11
Keer, L. -----	52
Keith, G. H. -----	18
Kennedy, E. M., Jr. -----	63
Kennel, B. -----	26
Kenney, R. A. -----	127
Kern, W. I. -----	26
Kerwin, R. I. -----	81
Kimball, K. E. -----	198
Kindall, J. V. -----	103
Kindley, L. M. -----	60
Kirchner, H. P. -----	101, 102, 111, 112
Klahs, L. J. -----	210, 219
Klaus, E. E. -----	130, 144
Klinger, R. F. -----	38
Klopp, W. D. -----	15, 69
Kober, E. H. -----	75
Kontrovitz, H. S. -----	227
Koo, R. C. -----	18
Koskinen, M. F. -----	39
Krause, H. H. -----	89
Kronenberg, K. J. -----	163
Ku, P. M. -----	138, 148, 149, 150
Kuchta, J. M. -----	133
Kulin, S. A. -----	24
Laid, R. I. -----	133
Lajiness, W. G. -----	92
Lamb, J. P. -----	150
Lang, J. I. -----	177, 193
Langlois, A. P. -----	215, 216
Lantz, C. D. -----	214
Lapple, C. E. -----	216
Lauchner, J. H. -----	101
LaVecchia, A. -----	21, 25
Lavik, M. T. -----	153
Lawler, J. H. L. -----	131, 137
Lawrie, W. E. -----	57
Lawthers, D. D. -----	65
Lazan, B. J. -----	52

	Page
Lesch, A. E. -----	230
Leathen, W. W. -----	230
Lebovits, A. -----	82
Leforge, J. W. -----	161, 177
Leininger, R. I. -----	88
Lemcoe, M. M. -----	96
Levine, N. -----	95
Levinestein, M. A. -----	27, 194
Lewis, D. W. -----	157
Libby, P. A. -----	176
Liggett, R. W. -----	123
Lindeneau, G. D. -----	126
Lis, S. J. -----	122
Lisanke, R. J. -----	79
Little, C. -----	119
Lloyd, R. D. -----	201
Lothrop, R. S. -----	161
Love, B. -----	16, 20, 198
Love, D. H. -----	216
Luck, J. -----	25
Luirette, A. J. -----	104
Lulling, R. M. -----	202
Luxon, J. T. -----	227
Lynch, C. T. -----	5
Lynn, A. L. -----	165
Lytton, J. L. -----	24, 84
 Machlan, G. R. -----	 109
Mack, E. B. -----	55
Macklin, B. A. -----	59
MacPherson, B. M. -----	54
Madorsky, S. L. -----	70
Mahoney, C. L. -----	151
Manchetti, J. G. -----	225
Manemeit, F. E. -----	210, 219
Manos, W. E. -----	169
Marino, A. -----	208
Maropis, N. -----	223
Marrs, O. L. -----	70
Marolo, S. A. -----	110, 114
Martin, A. -----	186
Martin, L. J. -----	139
Martin, M. -----	96
Marvel, C. S. -----	80, 89
Marynowski, C. -----	72
Mattice, J. J. -----	61
Maus, E. H. -----	63
Mayer, R. A. -----	88

	Page
Maykuth, D. J. -----	15, 69
Maziyasni, K. S. -----	16, 65
Meade, D. J. -----	37, 51
Mecklenburg, K. R. -----	139
Medved, T. -----	153
Mell, C. W. -----	139
Mellen, D. M. -----	119
Mentel, T. J. -----	34, 52
Merkl, B. A. -----	92
Merra, S. -----	129
Merrihew, F. A. -----	192
Mersol, S. A. -----	5
Metcalfe, A. G. -----	55
Meyers, C. L. -----	24, 84
Mickey, J. W. -----	81
Milek, J. T. -----	139
Mill, T. -----	72
Miller, C. D. -----	95
Miller, H. C. -----	67
Miller, J. E. -----	226
Miller, J. O. -----	229
Miller, P. D. -----	51
Miller, R. I. -----	210, 211
Mincher, A. L. -----	195
Minges, M. L. -----	205
Mistretta, A. L. -----	192
Moeller, T. -----	80, 90
Moon, D. P. -----	202
Moore, G. -----	74
Moore, H. R. -----	173
Morecombe, D. J. -----	48
Morris, C. F. -----	224
Morton, M. -----	80
Morse, W. G. -----	221
Moss, B. B. -----	228
Murphy, C. J. -----	75
Murray, J. F. -----	73, 215, 216
Murray, K. -----	97
Muvdi, B. B. -----	193
McAndrew, J. B. -----	201
McBride, R. J. -----	113
McCarthy, P. R. -----	141
McCauley, R. B. -----	32
McClintock, F. A. -----	40
McCloskey, A. L. -----	77
McClure, J. M. -----	41
McCollum, J. D. -----	181, 189

	Page
McConnell, B. D. -----	155
McCormick, S. -----	109
McCown, J. W. -----	226
McDevitt, N. T. -----	187
McDonald, P. H. -----	147, 150
McFarland, B. -----	116
McGrath, J. C. -----	124
McKinsey, C. R. -----	195
 Nakamura, H. H. -----	 59
Nakayasu, H. -----	81
Neary, J. K. -----	216
Neel, D. S. -----	178
Nelson, S. J. -----	225
Neuber, I. H. -----	21
Nevitt, T. D. -----	167, 185
Newman, H. W. -----	46
Newton, E. H. -----	128
Ng, C. W. -----	156
Nichols, G. M. -----	79
Nielsen, M. L. -----	83
Nilson, D. -----	117
Nudelman, H. B. -----	47
 O'Brien, M. -----	 81
Ogden, H. -----	15, 69, 192, 202
Oglesby, S. -----	176, 178
Olcott, E. -----	109
Olson, B. -----	50
Olson, J. C. -----	162
Olson, W. Z. -----	202
Olszewski, W. J. -----	135
Orofino, T. A. -----	81
Ortner, M. H. -----	222
Osborn, C. L. -----	145
Ott, E. -----	64
Owens, F. S. -----	94
 Paciorek, K. L. -----	 92
Paine, R. M. -----	3
Papagallo, A. -----	28
Papirno, R. -----	29
Park, B. J. -----	119
Parker, W. J. -----	179, 180
Pawlikowski, S. L. -----	213
Pears, C. D. -----	176, 178
Pearson, R. -----	211
Pemsler, J. P. -----	184
Pergament, H. S. -----	218

	Page
Perkins, G. D. -----	183
Peterson, G. P. ----- 113,	115
Pfeiffer, H. G. -----	165
Philipchuk, V. -----	54
Pickett, A. G. -----	96
Piirma, I. -----	96
Pisarenko, G. S. -----	33
Pletenik, A. -----	165
Plyler, E. -----	169
Pocs, E. -----	33
Popoff, C. ----- 137,	140
Popper, P. -----	123
Posakony, G. J. -----	58
Post, H. W. -----	75
Powell, C. -----	69
Powers, D. H. -----	127
Pratt, C. A. ----- 10,	65
Price, C. F. ----- 173,	174
Proffitt, C. E. -----	229
Pugh, J. W. -----	27
 Raab, G. -----	 78
Rabinowicz, E. -----	141
Radell, J. ----- 180,	181
Raisen, E. -----	172
Revere, A. -----	18
Riber, C. R. -----	190
Richmond, J. -----	169
Rietz, C. H. -----	1
Rivolta, B. -----	28
Robbins, D. L. -----	107
Robertshaw, T. L. -----	27
Robinson, A. R. -----	33
Robinson, C. F. -----	183
Rogers, W. G. -----	4
Rolling, R. E. -----	172
Rollins, C. T. -----	151
Rollins, F. -----	57
Rolston, J. A. -----	104
Rondeau, R. E. -----	178
Roper, R. S. -----	92
Rosenberg, H. M. -----	190
Rosenbery, J. W. -----	205
Ross, S. T. -----	12
Rosenberty, J. W. -----	66
Roth, W. L. -----	164
Rousseau, J. -----	135
Rusisill, J. E. -----	160

	Page
Rudner, B. -----	82
Rudy, J. -----	54
Rumpel, W. F. -----	97
Rushing, C. -----	85
Russell, J. A. -----	149
Ryan, M. T. -----	184
Sabroff, A. M. -----	222
Sadownick, E. M. -----	1
Sama, L. -----	65
Sara, R. V. -----	9
Schatz, E. A. ----- 59,	161
Schlar, C. B. -----	89
Schlogh, K. -----	85
Schmidt-Collerus, J. J. ----- 74,	183
Schmidt, D. L. ----- 110,	114
Schmidt, F. F. -----	15
Schmidt, R. -----	25
Schmitt, R. G. -----	62
Schmude, K. S. -----	172
Schnabel, W. J. -----	75
Schnitzel, R. H. -----	18
Schoenfeld, W. J. -----	220
Schoning, F. R. L. -----	160
Schroeder, H. A. -----	75
Schuette, E. H. -----	36
Schulman, S. -----	119
Schultz, R. L. -----	34
Schwartz, H. S. ----- 110,	118
Schwartz, M. M. -----	226
Schwartzbart, H. -----	54
Schwebke, G. L. -----	70
Schwenker, R. F. -----	126
Scott, H. F. -----	68
Seban, R. A. -----	172
Seldin, E. J. -----	8
Sell, H. G. -----	18
Sernka, R. P. -----	12
Sethna, P. ----- 49,	52
Sessoms, O. V. -----	167
Seyferth, D. -----	78
Shaffer, A. -----	135
Shank, M. E. -----	120
Sheehan, J. P. -----	47
Sheely, W. F. -----	195
Sheffer, D. E. -----	221
Shimmin, K. D. -----	31
Shinozuka, M. -----	43

	Page
Sidebottom, O. M. -----	32, 33, 35, 193
Sieron, J. K. -----	97, 98
Simcoe, C. R. -----	201
Simmons, P. A. -----	227
Simon, I. -----	10
Singleton, A. H. -----	66
Skramstad, H. -----	169
Sleven, R. -----	167
Small, R. H. -----	183
Smetana, F. O. -----	125
Smith, H. A. -----	91
Smith, H. E. -----	205
Smith, J. O. -----	132, 133
Smith, L. L. -----	100
Smith, R. K. -----	137, 140
Snyder, A. D. -----	210, 219
Spector, H. -----	105, 218
Speh, K. -----	167
Spolan, I. -----	133
Sprague, R. -----	7
St John, A. D. -----	144
Stafford, F. -----	2, 3, 6
Stair, R. -----	169
Stalego, C. J. -----	109
Standerfer, F. -----	211
Starks, D. F. -----	110
Stavrakes, E. J. -----	127
Stejskal, L. M. -----	204
Sterner, C. J. -----	66
Stewart, R. B. -----	158
Stokes, S. -----	76
Stonehouse, A. J. -----	3
Strauss, S. -----	70
Strnat, K. -----	163
Sugawara, K. F. -----	185
Susman, S. -----	103
Swan, G. A. -----	182
Swynnerton, N. F. -----	145
Sykes, G. E. -----	97
 Taft, M. -----	 116
Takamizawa, M. -----	78
Talley, C. P. -----	182
Tamborski, C. -----	74, 77, 184, 189
Taylor, D. E. -----	169
Taylor, G. R. -----	182
Taylor, R. -----	175
Taylor, W. E. -----	145

	Page
Temin, S. C. -----	82
Tewksbury, E. J. ----- 130,	144
Thomas, A. D. -----	151
Thomas, H. C. -----	229
Thomas, I. M. ----- 73,	82
Thomas, J. G. -----	223
Thomas, L. L. -----	162
Tiede, R. L. -----	109
Tierney, P. A. -----	157
Tietz, T. E. ----- 24,	84
Tombaugh, R. W. -----	14
Torbe, I. -----	40
Torborg, R. -----	25
Torrible, E. G. -----	82
Trepus, G. E. -----	92
Trop, D. C. ----- 140,	146
Troy, W. C. ----- 55,	227
Truell, R. -----	37
Turner, J. R. -----	221
Tuttle, M. M. -----	199
Tyler, J. C. -----	138
Underwood, C. R. -----	5
Vahldiek, F. W. -----	5
Van Deusen, R. L. -----	87
Vanik, M. C. -----	209
van der Burg, S. -----	225
Van Vliet, R. M. -----	59
Van Wazer, J. R. -----	83
Vasileff, N. -----	109
Vassallo, F. A. ----- 101, 102, 111,	112
Verhaegen, G. ----- 2, 3,	6
Vila, V. M. -----	117
Vogan, J. W. -----	110
Wacks, M. E. -----	70
Walker, W. R. -----	93
Wallouch, R. W. -----	8
Watanabe, H. -----	211
Weatherford, W. D. -----	138
Weber, H. S. -----	1
Weber, J. H. -----	74
Wechsler, A. E. -----	10
Weidman, R. ----- 223,	224
Welge, W. -----	134
Westbrook, J. H. -----	2
Whitfield, J. K. -----	147
Whittier, J. S. -----	46

	Page
Wilcox, B. A. -----	36, 38, 47
Willhelm, C. A. -----	42
Wilks, C. R. -----	226
Williams, D. N. -----	192
Williams, R. J. -----	92
Wilsdorf, H. G. F. -----	160
Wilson, H. H. -----	4
Wilson, J. L. -----	195
Wilson, R. -----	6
Wilson, W. A. -----	167, 181, 185, 189
Wineman, R. J. -----	83
Withers, J. C. -----	59, 161
Witt, E. R. -----	145
Wolf, R. A. -----	163
Wolfe, R. A. -----	163
Wolfe, R. P. -----	227
Wolfshagen, R. -----	134
Wood, D. L. -----	2
Wood, R. A. -----	192
Wood, W. W. -----	43, 199
Woods, W. G. -----	77
Woodward, R. D. -----	32
Wurst, J. C. -----	205
Wurster, C. F., Jr. -----	132
Yamazaki, H. -----	78
Yendall, E. F. -----	135
Yockey, H. P. -----	210
Young, W. R. -----	56
Yurenda, S. -----	103
Zabetakis, M. G. -----	133
Zuccarello, R. K. -----	126
Zung, V. T. -----	84

III. APPENDIX, CONTRACTOR INDEX

	Page
Advanced Materials Laboratory -----	8
Advanced Metals Research Corp. -----	16
Aerojet-General Corp. ----- 1, 107, 117,	210
Aerojet-General Nucleonics -----	211
Aeronutronics -----	12
Aeroprojects, Inc. -----	223
Agriculture, Dept. of -----	108
Airborne Instruments Laboratory -----	142
AiResearch Manufacturing Co. -----	135
Air Products, Inc. -----	66
Akron, University of -----	80, 96
Allied Chemical Corp. -----	68
American Cyanamid Co. -----	62
American Machine & Foundry Co. ----- 60, 122, 161,	170
American Oil Co. ----- 167, 181, 185,	189
Arizona, University of -----	89
Armour Research Foundation ----- 1, 47, 57, 59, 95,	100
	108, 172, 177, 201
Armstrong Cork Co. -----	187
Arthur D. Little, Inc. ----- 10, 128,	129
Atlantic Research Corp. -----	109
Atomics International -----	175
Automation Industries, Inc. -----	58
Battelle Memorial Institute ----- 5, 16, 41, 51, 69, 88,	89
	188, 192, 202, 222
Bell Aerosystems Co. ----- 192, 193,	220
Bell Aircraft Corp. -----	193
Bell & Howell Research Center -----	183, 190
Belfour Engineering Co. -----	197, 205
Bendix Products Division -----	110
B. F. Goodrich Co. -----	70
Birbeck College -----	73
Boeing Airplane Co. ----- 92, 93, 199, 204,	229
Brown University -----	37
Brush Beryllium Co. -----	2, 54
Brussels, University of -----	2, 6
B. S. A. Research Centre -----	17
Buffalo, University of -----	75
California, University of -----	44
California, University of Southern -----	125
Celanese Chemical Co. -----	145
Chance Vought Corp. ----- 43, 100,	197
Chicago, University of -----	174
Cincinnati Milling & Grinding Machines, Inc. -----	213
Cincinnati, University of -----	84

	Page
Clemson College -----	4
Clevite Corp. -----	226
Columbia University -----	34, 43, 48
Convair -----	32
Cornell Aeronautical Lab, Inc. -----	101, 111, 112
Crucible Steel Co. of America -----	11, 230
Cutler-Hammer, Inc. -----	167
Dayton, University of -----	66, 113, 205
Denver Research Institute -----	74, 183
Douglas Aircraft Co. -----	206, 228
Dow Chemical Co. -----	36, 91
E. I. DuPont -----	79
Electro-Optical Systems -----	173
Emerson & Cuming, Inc. -----	161, 177
Fabric Research Laboratories, Inc. -----	120, 121, 127
Florida, University of -----	71, 85
F&M Scientific Corp. -----	186
Forest Products Laboratory -----	113, 117, 198, 202
Franklin Institute -----	160
General Applied Science Laboratories, Inc. -----	218
General Electric Co. -----	2, 27, 56, 73, 86, 159, 164, 165 194, 229
General Dynamics -----	214, 215, 216, 221, 224
General Motors Corp. -----	227
Georgia Institute of Technology -----	61, 126
Goodyear Aircraft Corp. -----	229
Gulf Research & Development Co. -----	134, 141
Hamilton Standard Division -----	26
Harvey Aluminum, Inc. -----	14
Hexcel Products, Inc. -----	204
Honeywell Research Center -----	25
Hooker Chemical Corp. -----	79
E. F. Houghton & Co. -----	137, 140
Hughes Aircraft Co. -----	139
Illinois Institute of Technology -----	54
Illinois, University of -----	32, 33, 35, 80, 90, 193
Illinois University -----	101
Indiana Steel Products Division -----	163
International Telephone & Telegraph Corp. -----	208
Iowa State University -----	70

	Page
King's College -----	182
Koppers Co., Inc. -----	82, 90
Laboratori Di Electtrochimica -----	21, 23, 25, 28
Laboratories for Applied Sciences -----	169, 178
Lehigh University -----	147
Linde Co. -----	135
Lockheed Aircraft Corp. -----	24, 84
Marquardt Corp. -----	201
Martin Co. -----	22, 23, 97, 226
Massachusetts Institute of Technology -	39, 78, 119, 120, 141
Materials Research Corp. -----	45
Mellon Institute -----	81
Melpar, Inc. -----	60
Manufacturing Laboratories, Inc. -----	9, 11, 24
Michigan, University of -----	49, 196, 200
Midwest Research Institute -----	49, 139, 141, 144, 152 153, 154, 156
Minnesota, University of -----	30, 33, 34, 46, 49, 50, 52
Monsanto Chemical Co. -----	83, 132, 133
McGraw-Hill Book Co. -----	121
Narmco Industries, Inc. -----	103, 104
National Bureau of Standards -----	19, 70, 158, 169
National Carbon Co. -----	7, 8, 9
National Northern Division -----	54
New York, University of -----	20, 29, 30
North Carolina State College -----	147, 150
Northrop Corp. -----	44, 105, 218
Nuclear Metals, Inc. -----	184
Ohio State University -----	32, 39
Olin Mathieson Chemical Corp. -----	75, 219
Western Ontario, University of -----	82
Owens-Corning Fiberglas Corp. -----	109
Pennsalt Chemicals Corp. -----	67
Pennsylvania State University -----	130, 144
Plasmadyne Corp. -----	116, 137
Polytechnic Inst. of Brooklyn -----	171, 175, 176
RCA Laboratories -----	159
Rensselaer Polytechnic Institute -----	156
Republic Aviation Corp. -----	170
Research Chemicals -----	16, 162, 198
Rocketdyne -----	148

	Page
Shell Development Co. -----	151
Solar Aircraft Co. -----	55, 227
Southampton, University of -----	37, 40, 87
Southern Research Institute -----	42, 123, 124, 176, 178
Sperry Gyroscope Co. -----	143
Southwest Research Institute--	68, 96, 138, 148, 149, 150, 160
Stanford Research Institute -----	72, 116
Sylvania Electric Products -----	65, 213
Systems Research Laboratories, Inc. -----	22
Technische Hochschule -----	21, 26
Texaco Experiment, Inc. -----	182
Textile Research Institute -----	126
Thiokol Chemical Corp. -----	95, 212, 221
Thompson Products, Inc. -----	131, 132
Thompson Ramo Wooldridge -----	63, 64, 134
Union Carbide Corp. -----	6
Union Carbide Metals Co. -----	20, 195
Universal-Cyclops Steel Corp. -----	220
U. S. Borax Research Corp. -----	77
U. S. Department of the Interior -----	133
U. S. Naval Radiological Defense Laboratory -----	179, 180
U. S. Naval Research Laboratory -----	76
U. S. Rubber Co. -----	225
Utah, University of -----	4, 151
Vienna, University of -----	85
Vitro Corp. -----	223, 224
Western Gear Corp. -----	146
Westinghouse Electric Co. -----	14, 18, 48, 157, 158
W. R. Grace & Co. -----	209
Wyandotte Chemical Corp. -----	61, 92, 136

Aeronautical Systems Division, Dir/Materials and Processes, Deputy for Technology, Wright-Patterson AFB, Ohio.
Report Nr WADC TR 53-373, Suppl 9. A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM. Final report, Nov 62, 250 p.

Unclassified report

These reports cover basic and applied research in the materials area being conducted by the Metals and Ceramics, Non-Metallic Materials, Physics, Manufacturing Technology, and Applications Laboratories of the Directorate of Materials and Processes.

(over)

- I. Materials Research, Basic, Applied
- I. Donna J. Tate
- II. Aval fr OTS
- III. In ASTIA collection

Aeronautical Systems Division, Dir/Materials and Processes, Deputy for Technology, Wright-Patterson AFB, Ohio.
Report Nr WADC TR 53-373, Suppl 9. A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM. Final report, Nov 62, 250 p.

Unclassified report

These reports cover basic and applied research in the materials area being conducted by the Metals and Ceramics, Non-Metallic Materials, Physics, Manufacturing Technology, and Applications Laboratories of the Directorate of Materials and Processes.

(over)

- I. Materials Research, Basic, Applied
- I. Donna J. Tate
- II. Aval fr OTS
- III. In ASTIA collection